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Quality Assurance Plan: Love Canal Study
Appendix Q. Subcontractors' QA Plans

GCA Corp.
Bedford, MA

Prepared for

Environmental Monitoring Systems Lab.
Research Triangle Park, NC

Dec 82

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December 1982

QUALITY ASSURANCE PLAN

LOVE CANAL STUDY

LC 1-619-026

EPA CONTRACT 68-02-3168

APPENDIX Q

SUBCONTRACTORS' QA PLANS

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16. ABSTRACT

The four volumes in this set comprise the working guideline documents for the Love Canal Study. The documents were developed to direct both the prime contractor and subcontractors while performing for the Environmental Protection Agency. Detailed procedures for each analysis type are described. Procedures for all field sampling are also described. Quality assurance plans submitted by each subcontractor and approved by the prime contractor are provided. A description of the document control procedure, chain-of-custody procedure, various quality control procedures, and other study operational procedures are provided.

Together the four documents provide a "work plan" for the Love Canal effort. Since the volumes were developed prior to the actual field sampling, no environmental results are included.

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APPENDIX Q

SUBCONTRACTORS' QA PLANS

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1.0 PROJECT DESCRIPTION AND ORGANIZATION

1.1 PROJECT DESCRIPTION

An area, called the Declaration Area, around Love Canal in Niagara Falls, New York, has been declared a Federal Emergency Area. To determine the health hazard to residents of that area the Environmental Protection Agency has designed an integrated multimedia study which includes sampling and analysis of air, water, soil, sediment, and biota in the area. This information will be used in part to estimate the extent identified substances contribute to possible increased risks encountered by residents.

1.2 PROJECT ORGANIZATION

GCA/Technology Division is under contract to the Environmental Protection Agency to conduct the Love Canal study described above; Steven Bromberg, EMSL/RTP, is the EPA Project Officer. In order to expedite the program in a timely manner, GCA is conducting the sampling, analysis, and geotechnical efforts through selected subcontractors under the direction of GCA senior technical staff members. Table 1.2.1 lists the subcontractors and their respective project activity.

1.3 SUBCONTRACTORS' QA PLANS

Each subcontractor is required to submit to GCA a QA Plan specific to his project activity. GCA's QA Manager has provided to each subcontractor a suggested QA Plan outline with format suggestions and information on Document Control and Chain-of-Custody procedures.

The submitted QA Plans have been reviewed, revisions requested when necessary, and incorporated in this Appendix. Particular care has been given to ensuring that EPA's internal QC requirements, submitted as part of the subcontract documents, have been met by the subcontractors' QA Plans. The plans are grouped according to project activity, as in Table 1.2.1.

Due to the different heading formats used by subcontractors, and the fact that some revisions and QA Plans have not yet been received, this appendix is not page-numbered. Instead, each QA Plan is assigned an Appendix Q Plan number; this number will be found on the upper right-hand corner, or the right center portion, of each page. Subcontractor headings have been retained in all cases. The Contents Section lists the major Appendix Q Sections and the QA Plans contained in each section.

TABLE 1.2.1 LOVE CANAL STUDY SUBCONTRACTORS AND PROJECT ACTIVITIES

Subcontractor	
	Sampling/Analysis Preparation
PEDCo Environmental, Inc.	Preparation of Tenax cartridges
Southwest Research Institute	Preparation of polyurethane foam plugs
Research Triangle Institute	Spiking of Tenax cartridges
IIT Research Institute	Verification of spiked Tenax cartridges
	Sample Collection
Geomet Technologies, Inc.	All except groundwater-related
JRB Associates, Inc.	Groundwater-related
	Sample Analysis
Battelle Columbus Laboratories	Volatile organics on Tenax
PEDCo Environmental, Inc.	Volatile Organics on Tenax
Gulf South Research Institute	Pesticides on polyurethane foam; organics in water, soil and sediments
Southwest Research Institute	Pesticides on polyurethane foam; soil and sediments methods study; organics in soil, sediments and biota; inorganics in water, soil, sediments and biota.
Compuchem/Mead Technology Labs	Organics in water, soil, and sediments
PJB/Jacobs Engineering Group, Inc.	Organics in water, soil, and sediments; inorganics in water, soil, and sediments
TRW, Inc.	Organics in water
Acurex Corporation	Soil and sediments methods study; organics in water, soil, and sediments
Energy Resources Company, Inc. (ERCO)	Inorganics in water, soil, and sediments
Midwest Research Institute	Organics in biota
Wright State University	Dioxin in all media
Advanced Environmental Systems, Inc.	TOC/TOX, pH, conductivity in groundwaters
Wadley Institutes of Molecular Medicine	Interferon in mice
	Geotechnical Study
JRB Associates, Inc.	Supervisory geologist-interpretation of existing geotechnical information, coordination of drilling program, geologic logging, etc.
Empire Soils Investigations, Inc.	Well drilling
Technos, Inc.	Geophysical surveys
GeoTrans, Inc.	Geophysical modeling

2.0 SAMPLING/ANALYSIS PREPARATION

The subcontractors involved in the preparation of air collection media and the preparation and verification of QA/QC Tenax samples are listed below. As shown in Table 1.2.1, PEDCO Environmental, Inc. and Southwest Research Institute are also analytical subcontractors; their QA Plans are included in Section 4.0 of this volume.

Subcontractor	Project Activity
PEDCO Environmental, Inc.	Preparation of Tenax cartridges
Southwest Research Institute	Preparation of polyurethane foam plugs
Research Triangle Institute	Spiking of Tenax cartridges
IIT Research Institute	Verification of spiked Tenax cartridges

The suggested outline provided to these subcontractors was the same as that presented to the analytical subcontractors; it is shown here on the following page of this section.

SUBCONTRACTOR'S QA PROJECT PLANS FOR ANALYSIS OF SAMPLES FROM LOVE CANAL

Outline

1.0 Project Description and Organization

2.0 Major Project Elements

3.0 QA Objectives - State expected precision and accuracy to be achieved by each analytical method in each media analyzed.

4.0 Document Control Procedures - Overall framework, numbering system, sample tags, Custody Forms, Log Books, Data Sheets provided by Prime Contractor. State your internal operation of the system, identifying responsible person as Document Control Officer.

5.0 Chain of Custody - Same as 4.0.

6.0 Analytical Internal QC Checks - Provide a table for each media (air, water, etc.) analyzed which lists operational checks for each analytical method. Control limits for each operational check should be provided with the corrective action to be taken if control limits are exceeded.

7.0 Data Reporting Management System - Standardized data forms will be provided by Prime Contractor - both analytical data sheets and encoding forms for the data management system. Internal operation of the system, and data review before reporting should be described.

8.0 Corrective Action Procedures - State how corrective actions will be implemented in the laboratory - identify responsible personnel. The corrective actions should have been identified in Section 6.0.

Appendix B Analytical Procedures

Part 1. Analytical Methods. Provided by Prime Contractor.

Includes analytical data sheets. Calibration of sampling equipment forms part of operating procedure--subcontractor provides for his equipment, unless given in analytical method.

Part 2. Analytical Equipment Maintenance/Troubleshooting Procedures

- GC/MS
- GC/ECD
- Analytical Balances
- AA--Cold Vapor Hg, Furnace Techniques
- pH and Selective Ion Meters
- Technicon Autoanalyzer, If Used
- Other Equipment

IIT Research Institite QA Plan

QA PROJECT PLAN FOR ANALYSIS OF QA/QC SAMPLES FROM LOVE CANAL

1. PROJECT DESCRIPTION AND ORGANIZATION

IIT Research Institute (IITRI) has been subcontracted by GCA/Technology Division (GCA) to perform quality assurance checks on ambient air samples taken in the Love Canal area of Niagara Falls, NY. Specifically, the subcontract requires the quantitative analysis of nine QA/QC Tenax cartridge samples for nine organic compounds as well as perfluorobenzene and octafluorotoluene, and the qualitative identification of chloroform, if present. Characterization and quantification of the components in the cartridges are carried out by high-resolution gas chromatography/mass spectrometry/data system. Appropriate standardization and quality control measures will be performed as required.

The nine samples will be delivered in three batches of three samples each. Notification of sample shipment to IITRI will be verbally provided by GCA, ten days before the expected date of arrival. IITRI will complete analysis, including the generation of appropriate calibration data in triplicate for each sample set, within seven days of sample receipt.

2. QA OBJECTIVES: PRECISION AND ACCURACY

The reproducibility of the method using sorbent samplers depends on several factors. Using the cyclic scan mode and replicate samples, precision is expected to range from ±10% to ±30% of the relative standard deviation for different substances. Analytical accuracy is expected to be ±10% to ±30% depending on the nature of the compound.

3. DOCUMENT CONTROL PROCEDURES

Overall Framework

The sample custodian at IITRI receives the samples and, after inspecting them and satisfying himself that they have not been tampered with, he implements a system to maintain control of the samples, from receipt through reporting of the analytical results.

Numbering System

Upon recipt of the samples, each sample is identified and numbered by means of a permanently affixed label which indicates the source of the sample and the date of recipt. Each sample code is also entered into a project sample log book, using the information on the chain-of-custody sheet which accompanies each sample.

The sample numbering system used consists of a project number, consecutive sample identification number and a code indicating the method of analysis being used. A typical sample identification number is the following:

C6999 - 0001 - GCMS

where C6999 is the assigned IITRI project number, 0001 is the consecutive sample identification number, and GCMS is the analytical technique used. Information relating to the sample is recorded in a subsample list as:

0001 - GCMS 0001 - HPLC

In this example, the GC-MS and HPLC laboratories record all necessary information in their respective project logbooks, and on completion of the analyses, the results are reported using the original sample identification number, project logbook number and page numbers. A description of the sample identification, project logbook and subsample list format is as follows:

SAMPLE LOT[1] NUMBER	SAMPLE IDENTIFICATION[2] NUMBER OR DESCRIPTION	DATE SAMPLE RECEIVED	DATE SAMPLE LOGGED	DISPOSITION OF UNUSED PORTION OF SAMPLE

Subsample List Begins on Page _____

1. Assigned by DCO using project number and four digit consecutive number.
2. Sponsors identification number or description, or specific identification for a sample assigned by project personnel.

CONSECUTIVE NUMBER	SUBSAMPLE NUMBER OR METHOD	PROJECT LOGBOOK NUMBER	PAGE	ANALYSIS FOR	OTHER

Sample label:

DATE:

PROJECT NUMBER	CONSECUTIVE NUMBER	SUBSAMPLE NUMBER

SAMPLE RECEIVED FROM:

Data Handling and Reporting

All raw data are recorded in ink directly in prenumbered IITRI logbooks or data forms that are bound on conclusion of the project. Original laboratory data, calculations, sketches, etc., are entered directly into the logbook. Certain material generated in the course of the project will, of necessity, be external to the logbook. However, logbook entries are made in diary fashion when performing such work. Witnessing is done at least once a month by personnel who have not performed the work recorded, but who are competent to understand the technical aspects. The Document Control Officer (Mr. Kirit Parihk) reviews and approves all laboratory data before they are forwarded to Main Files. Written reports are submitted on Data Sheets provided by GCA.

4. LABORATORY CHAIN-OF-CUSTODY

The Document Control Officer (DCO) takes custody of the shipped samples and checks the sample tag information against the custody records. He also checks the sample condition and notes anything unusual under "Remarks" on the custody form. The DCO then enters the sample tag data into the project sample identification logbook. If a discrepancy is found between a sample tag number and the custody record listing, the DCO attempts to resolve the problem by checking all available information. The GCA QA Manager will be notified of discrepancies by telephone, followed by written notification. Such samples will be held in storage and will not be analyzed until the problem has been satisfactorily resolved by contacting the GCA QA Manager or other designated responsible authority.

All samples will be stored at 4°C until analyzed, in a refrigerator located in a locked area. All logbooks will also be stored in a locked area.

The DCO hands over custody of each sample to the GC-MS personnel for analysis. The name of the individual who receives the sample is recorded in the logbook and entered on the sample custody record. The laboratory personnel are responsible for the care and custody of the samples from the time of receipt until they are analyzed.

Once the analyses and appropriate quality assurance checks have been carried out, the unused portion of each sample will be disposed of in accordance with the direction of the GCA QA Manager. All identifying tags, data sheets, and laboratory records will be retained as part of the permanent documentation record.

5. ANALYTICAL INTERNAL QC CHECKS

Pellizzari has defined the minimum performance specifications acceptable for ambient air analysis, using high-resolution GC and a magnetic-sector mass spectrometer.

Mass calibration and intensity, as well as chromatographic performance of the GC-MS-COMP system, are evaluated by using a system performance mixture which is loaded onto Tenax cartridges.

The system performance mixture contains perfluorobenzene, perfluorotoluene, ethylbenzene, p-xylene, 5-nonanone, acetophenone, 2,6-dimethylaniline, and 2,6-dimethylphenol.

Mass and intensity calibrations are performed using perfluorokerosene (PFA-225US). The perfluorotoluene in the performance mixture serves to monitor instrument stability (high/low mass balance) in terms of mass resolution and relative ion abundance under GC operating conditions. Table 1 lists the tolerances for the major peaks which the mass spectrometer must meet.

Chromatographic performance is evaluated according to the criteria listed in Table 2. Corrective action must be taken if any of the parameters in Table 2 fall outside the limits indicated. Corrective action must also be taken if the observed concentration of any component, based on the original calibration, from a Tenax cartridge standard is greater than ±20% of the actual concentration. Corrective action could include retuning of the mass spectrometer verification of capillary column performance, or verification of the cleanliness of the system by running a blank cartridge. If the analysis of a second cartridge representing the same concentration still shows concentrations which are greater than ±20% of the actual concentration, based on the original calibration curve, a new calibration curve must be generated before further samples are analyzed.

Table 1

MASS AND INTENSITY TOLERANCES ACCEPTABLE FOR CALIBRATION OF MAGNETIC INSTRUMENTS FOR QUANTITATION OF AMBIENT AIR SAMPLES

Perfluorotoluene	
m/z	%I (C.V.)
69	34 (5)
79	6 (10)
93	15 (8)
117	35 (8)
167	14 (7)
186	63 (6)
217	100 (0)
236	86 (4)

Table 2

SPECIFICATIONS OF PERFORMANCE FOR GLASS CAPILLARY COLUMN[a]

Parameter	Test Compound(s)	Value ± S.D. (C.V.)
Resolution[b]	Ethylbenzene:p-Xylene	1.30 ± 0.11 (8)
% Peak Asymmetry Factor[c]	Nonanone	130 ± 32 (25)
	Acetophenone	260 ± 34 (13)
Acidity[d]	2,6-Dimethylaniline:Acetophenone	1.00 ± 0.10 (9)
Basicity[e]	2,6-Dimethylphenol:Acetophenone	0.80 ± 0.03 (5)

a. SE-30 capillary column.
b. $R = 2\Delta W/(W_1 + W_2)$, where ΔW = retention time difference, W = peak width at base.
c. %PAF = (B/F) x 100, where B = area of back half of peak, F = area of front half of peak.
d. Acidity = (Peak area of dimethylaniline)/(Peak area of acetophenone).
e. Basicity = (Peak area of dimethylphenol)/(Peak area of acetophenone).

6. DATA REPORTING MANAGEMENT SYSTEM

All data are entered into prenumbered IITRI logbooks. After the data and calculations have been checked and approved by the laboratory supervisor, they are submitted to GCA by telephone. Thereafter, they are recorded on standardized data sheets which are sent to GCA.

7. CORRECTIVE ACTION PROCEDURES

Corrective action procedures are implemented as required, once the nature of the problem requiring correction has been identified. As pointed out in Section 5, these may include measures associated with the mass spectrometer itself (ion source, detector, electronics, vacuum malfunction), the gas chromatograph (column, flow, temperature malfunction) or the data system (hardware, software malfunction). Corrective action procedures are normally invoked by the responsible personnel in a careful, systematic manner. The responsible personnel are Ms. Louise Brousek (Assistant Chemist, GC-MS Operator); Mr. Don Krystof (Assistant Chemist, Data System Operator); and Dr. Sydney Gordon (Senior Chemist and Head, Mass Spectrometry Laboratory).

APPENDIX I (As per Work Scope, 10/15/80)

APPENDIX B. ANALYTICAL PROCEDURES

1. ANALYTICAL PROTOCOL: VOLATILE ORGANICS ON TENAX

A. SUMMARY OF METHOD

Concentrations of selected volatile organic compounds, listed in Attachment I, which are loaded onto Tenax^R sorbent cartridges, are to be measured for quality assurance purposes. The volatile organic constituents are determined by thermal desorption of the Tenax^R cartridges such that the desorbed organics are first cryogenically trapped and then flashed onto a capillary column for GC/MS analysis.

B. APPARATUS AND MATERIALS

1. *Desorption Equipment*

- Thermal desorption and trapping apparatus (Nutech Model 320 Thermal Desorption System)
- Mass flow controller (Brooks Model 8744A), calibrated for He in the 0-10 cc/minute range
- Liquid nitrogen
- Ultrahigh purity helium
- Vacuum dewar
- Bubble Meter

2. *Gas Chromatograph*

- Analytical gas chromatographic system (Varian 2740) with capillary inlet (Hewlett-Packard Model 18835A) and oven temperature programming capability, and amenable to interface with the above thermal desorption equipment.
- Capillary column: 41 m. SGE SE-30, SCOT, 0.5 mm I.D. column (0.8-2.0 μ film thickness).

3. *Mass Spectrometer*

- Mass spectrometer (Varian MAT 311A sector-magnet) interfaced to the above gas chromatograph and to a data system (Varian MAT SpectroSystem SS-200) with capabilities conforming to the specifications given in Method 624 Part 4.9, page 69532, Federal Register, Monday, December 3, 1979.
- Magnetic tape storage capability
- NIH/EPA library
- Software with a minimum capability of real-time acquisition of data, data manipulation and library searching.

The Varian MAT 311A mass spectrometer features a double-focusing analyzer with reverse Nier-Johnson geometry, and a differentially pumped vacuum system with high-speed pumping provided by two turbomolecular pumps. The system also offers high resolving power (>25,000, 10% valley definition), wide mass range (1-3600 a.m.u.), and a high-gain 16-stage Be-Cu secondary electron multiplier. The ion source consists of a combination standard electron impact source and an electron impact ionization detector (EID). By operating the EID at a suitably low electron energy, the carrier-gas-free total ion current analog signal from the GC is obtained directly in real-time using a strip-chart recorder.

The Varian MAT SpectroSystem SS-200/M, a state-of-the-art data system, is designed specifically for the acquisition and processing of mass spectral data, and features independent and simultaneous data acquisition and evaluation (from up to four mass spectrometers and/or four terminals), as well as sophisticated and extensive application-oriented software. The system in use at IITRI comprises a Digital PDP 11/34 computer with 128 Kwords (16-bit) memory, a 5M word disk unit, two Tektronix 4010 display terminals, a Tektronix 4631 hardcopy unit, a Datum/Tandberg magnetic tape system, and a Printronix P300 line printer/plotter.

Besides the standard applications software program package for data evaluation and presentation, a suite of programs has been developed to enhance GC resolution automatically and obtain "clean" spectra by applying a tabular peak modeling technique to mass chromatograms in the original data file.

C. CALIBRATION AND PERFORMANCE CRITERIA

1. *Instrument Tuning*

Mass spectrometer performance criteria are those set forth by Pellizzari and co-workers (EPA Contract No. 68-01-3849, RTI/1521/00-26S; Work Plan [Phase I]) for analysis of vapor-phase organic compounds in ambient air using magnetic-sector instruments. Performance specifications must be met daily or for every 8 hours of analysis time, and a permanent record of each performance check maintained.

2. *Capillary Column Performance*

Capillary column performance will be evaluated with "Grob" (acetophenone, 2,6-dimethylphenol, 2,6-dimethylaniline, nonanone, ethyl benzene and para-xylene) mixture spiked onto calibration and quality control checking cartridges. The following criteria must be met to ensure adequate capillary column performance:

(1) $\text{RESOLUTION} = \dfrac{2\Delta W}{W_1 + W_2} = 1.20 \pm 0.11$

where ΔW = Difference in retention times between ethylbenzene and p-xylene

W_1, W_2 = Average base width of peaks measured at the base

(2) PERCENT PEAK ASYMMETRY FACTOR (PAF) $\%\ \text{PAF} = \dfrac{B}{F} \times 100$

where B = Area of back half of peak

F = Area of front half of peak

Both measured at 10 percent above baseline

% PAF for acetophenone = 260 ± 34

% PAF for nonanone = 130 ± 32

(3) ACIDITY TEST (A.T.) = 1 ± 0.1 $A.T. = \frac{PAWB}{PAA}$

where PAWB = Peak area of dimethylaniline

PAA = Peak area of acetophenone

(4) BASICITY TEST (B.T.) = 0.8 ± 0.03 $B.T. = \frac{PAWA}{PAA}$

where PAWA = Peak area of dimethylphenol

PAA = Peak area of acetophenone

3. *Thermal Desorption System*

Proper operating temperatures and gas flow rates must be verified for at least each 8 hours of operation.

4. *Calibration*

Before initiation of sample analyses, a calibration curve in triplicate will be generated for perfluorobenzene and octafluorotoluene using standard Tenax^R cartridges and a vaporization system.

D. ANALYTICAL PROCEDURES

The Tenax cartridges are analyzed by thermal desorption at 250°C for 10 minutes in thermal desorption system. The desorbed organics are cryogenically trapped and subsequently flashed onto the specified glass capillary column. The effluent from the capillary is directly introduced into the mass spectrometer for analysis. The thermal desorption system manufacture's operating manual is followed to perform this sequence of operations.

Data Acquisition:

The following GC conditions are to be used for analysis:

Injector temperature 200°C

Column Oven Conditions: Initial temperature - 25°C, Hold 5 minutes, Ramp - 4°C/min, Final temperature - 200°C, Hold 15 minutes

Approximate flow: 2 cc/min

The mass spectrometer must be operated to accommodate the narrow peak widths produced by the capillary column. For this reason the following mass spectrometer conditions are to be used:

Source temperature: 250°C

Source base pressure: $<1 \times 10^{-6}$ torr; 4×10^{-5} torr with GC effluent entering

Scan time: 2 sec

Mass range: 35 to 400 amu

Electron energy: 70 eV

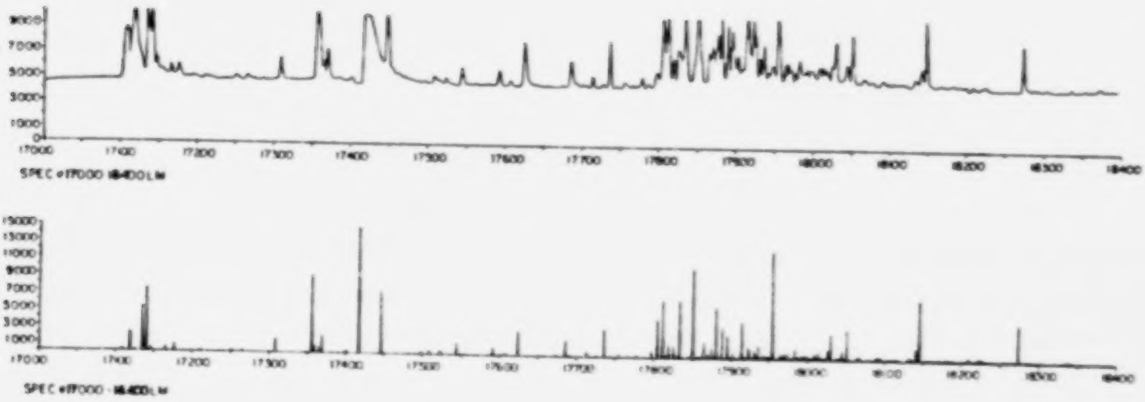
Filament emission: 1000 μA

Resolving Power: 700 (10% valley definition)

Ion abundance data from each scan are available in real-time as a function of spectrum scan number (or retention time). Total ion current profiles (TICP), extracted ion current profiles (EICP), or normalized mass spectra may then be examined individually using the stored GC-MS data. (Usually a TICP closely resembles the flame ionization detector chromatogram. An EICP is a plot of the variation in relative abundance of one or more ions as a function of time, and aids in locating specific compounds, or compound classes, in computer-operated TICPs). The immediate availability of the TICP plots is useful in deciding whether a particular run should be processed further or discarded.

Data Enhancement and Spectrum Identification

Before attempting to identify and quantitate individual components in complex mixtures, the raw GC-MS data files are routinely processed by means of a sophisticated data enhancement algorithm. Program CLEANUP automatically produces a file of resolved elutant spectra, free of background contributions, by the application of a tabular peak-modeling technique to mass chromatograms in the original data file. The resolved spectra may then be identified by means of a Biemann search, which is a routine that permits the sequential comparison of each unknown spectrum to standard spectra in a library. Compounds which do not yield acceptable identifications in this way are sought manually by comparing their spectra with published compendia of mass spectral data. The figure shows a typical TICP from an ambient air sample with the corresponding profile after application of the CLEANUP algorithm.



Total ion current profile (TICP) for an ambient atmosphere sample: (a) raw GC-MS data; (b) GC-MS data after application of CLEANUP algorithm.

Quantitation

For purposes of the present study, with the concurrence of Dr. Joe Bumgarner (EPA, EMSL/RTP), the data will be quantitated in the following way.

Calibration curves will be established in triplicate for perfluorobenzene and octafluorotoluene using the data obtained from standard Tenax cartridges. The amounts (ng per tube) of perfluorobenzene and octafluorotoluene present in each sample tube will be determined by comparing their responses to the calibration curves.

Relative concentrations of the remaining compounds designated for quantitation (as per Attachment I) will be determined with respect to the perfluorobenzene and octafluorotoluene present. After establishing the relative molar response factors for each compound of interest in separate experiments, using standard Tenax cartridges, the concentration of these compounds in the QA/QC samples will be calculated from their relative concentrations and response factors.

Quantitative estimation of specific components by GC-MS is not always feasible using only the total ion current value since baseline resolution between peaks is not always achieved. One way of dealing with this problem is to use reconstructed mass fragmentograms (i.e., single ion plots) for any suitable combination of m/e ions in the full mass spectra using available computer software programs. The approach adopted at IITRI utilizes the data obtained from the CLEANUP program which, in most cases, yields fully resolved spectra. Relative concentrations of the individual components of interest are then computed, based on one or more internal standards.

Relative concentrations of individual components are determined from the ratio of the peak total ion current (TIC) of the unknown to the peak TIC of the internal standard, i.e., for the ith component.

$$\text{Rel. Conc. (i)} = \frac{\text{Areal TIC of ith component}}{\text{Areal TIC of internal standard}} \qquad (1)$$

(The corrected peak TIC for each peak is obtained from program CLEANUP.)

The relationship between relative concentration and actual amount of material present in a sample must be established by auxiliary methods. Pellizzari has outlined an approach to obtain quantitative data using relative molar response (RMR) factors. This method, which obviates the need to obtain complete calibration curves for each compound for which quantitative information is desired, requires information on the exact amount of a standard added and the relationship of the RMR of the unknown to that of the standard.

The relative molar response factor is defined as:

$$\mathrm{RMR}_{un/std} = \frac{(\text{Areal TIC})_{un}/\text{moles}_{un}}{(\text{Areal TIC})_{std}/\text{moles}_{std}} \tag{2}$$

To determine the RMR, a known concentration of standard and unknown must be used to measure the system response in independent replicate experiments. The areal TIC of a component is a direct function of its concentration, so that Equation 2 may be rewritten in terms of the sample weight (g), in grams, and the gram molecular weight (MW) of the component:

$$\mathrm{RMR}_{un/std} = (\text{Rel. Conc.})_{un/std} \cdot (g_{std}/\mathrm{MW}_{std}) \cdot (\mathrm{MW}_{un}/g_{un}) \tag{3}$$

Once RMR factors have been established, the amount of each unknown component of interest present in a sample is found from:

$$g_{un} = \frac{(\text{Rel. Conc.})_{un/std} \cdot g_{std} \cdot \mathrm{MW}_{un}}{\mathrm{MW}_{std} \cdot \mathrm{RMR}_{un/std}} \tag{4}$$

(For the purposes of the present study, g_{std} is found from the calibration curves for perfluorobenzene and octafluorotoluene.)

It is important to note that the use of the CLEANUP program in this way permits the utilization of peak TIC values, which provide improved precision since they are generally much larger than the ion current values of individual fragment ions. Moreover, both qualitative and quantitative information is obtained in the same analysis by this approach, since the sample is first characterized from the data stored on disk or magnetic tape, and quantitation may be carried out at a later time. In those cases where peaks are incompletely resolved by the CLEANUP program, use is then made of suitable characteristic ions.

The minimum quantifiable limit has been determined to be 50 ng per compound per tube. We shall define our limit of detection for each component to be identified as a signal-to-noise ratio of 4:1, and report this value in ng per tube.

Components present at concentrations between this minimum detection limit and the minimum quantifiable limit are to be reported as "trace". The compound(s) designated for qualitative analysis shall be reported as "present" or "not detected" as appropriate. In addition all spectra collected are to be archived on magnetic tape in the specified format. Results shall be reported on the modified, analytical data sheets provided by GCA, copies of which are attached.

E. QUALITY CONTROL PROCEDURES

Procedural Control

A strict analysis sequence is followed. After mass and intensity calibration of the mass spectrometer has been achieved, a Tenax cartridge loaded with the performance mixture (cf. 5.0 Analytical Internal QC Checks) is first analyzed, to monitor the capillary GC column performance and the tuning of the MS. The performance mixture is followed by a blank, a calibration standard, an RMR standard mixture, and then up to 4 sample cartridges are analyzed, before the cycle is repeated. At the start of each working day, the analysis sequence begins with the performance mixture, the blank and standards, followed by the samples.

Corrective Action Measures

Corrective action must be taken if the observed amount of any component, based on the original calibration, from the desorbing standard cartridge is greater than ±20 percent of the actual amount. Corrective action could include retuning of the mass spectrometer, verification of capillary column

Appendix Q
Plan 2.2

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LOVE CANAL MONITORING PROJECT

COMPLETED BY: ____________

ANALYSIS DATA REPORTING FORM
INTERNAL QC REPORT

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80

SAMPLE ID	MEDIUM	METHOD	LAB. ID	DATE MO. DA.	TIME HR. MN.	SAMPLE SIZE
	AIR					

TENAX CARTRIDGES - QC DATA
- Laboratory Control Standard

	POLL. CODE	OBSERVED CONC.	EXPECTED CONC.	SPECIFIC METHOD	INT. STD. LCS	SURROGATE SPIKE	COMMENTS	JUL. DATE	CARD TYPE	SEQ. NO.
Benzene									A2	001
Carbon Tetrachloride									A2	002
Chlorobenzene									A2	003
O-Chlorotoluene									A2	004
1,2-Dibromoethane									A2	005
O-Dichlorobenzene									A2	006
1,1,2,2-Tetrachloroethylene									A2	007
1,2,4-Trichlorobenzene									A2	008
Toluene									A2	009
Hexafluorobenzene									A2	010
Octafluorotoluene									A2	011
									A2	012
Chloroform (Qualitative Identification)									A2	013
									A2	014
									A2	
									A2	
									A2	
									A2	
									A2	

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LC 1-619-026- ☐☐-J41-☐☐☐☐-☐

LOVE CANAL MONITORING PROJECT

COMPLETED BY: ____________

ANALYSIS DATA REPORTING FORM
COMMENTS

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80

SAMPLE ID	MEDIUM	METHOD	LAB. ID	DATE MO. DA.	TIME HR. MN.	SAMPLE SIZE
			▨			▲

COMMENTS

COMMENTS	JUL. DATE	CARD TYPE	SEQ. NO.
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	
	▨	A 4	

8/80

performance, or verification of the cleanliness of the system by running a blank cartridge. A second cartridge representing the same concentration can then be thermally desorbed and analyzed. If the observed concentration is still greater than ±20 percent of the actual concentration, based on the original calibration, a new calibration must be generated before any samples are analyzed.

In addition to the above routine quality control procedures, constant monitoring of the analyses for cross contamination must be established. This can be accomplished by analyzing blank Tenax cartridges provided, as well as analyzing a blank cartridge immediately after a sample has been analyzed which is heavily loaded with organics. A careful record of the sequence in which these blank cartridges is run must be kept. The samples themselves are to be run in the sequence in which they are numbered when they are received from GCA.

F. SAMPLE HANDLING AND PRESERVATION

The samples are received as part of a chain-of-custody sequence which must be maintained by the analytical laboratory. The samples are to be kept at 4°C until actually analyzed. The cartridges should not be removed from the sealed culture tubes until the time of thermal desorption. The cartridges must not be touched with bare hands; cloth gloves must be worn or appropriate tongs used to handle the cartridges. All thermally desorbed cartridges are to be stored in their original culture tube container until notified of their disposition.

2. MAINTENANCE/TROUBLESHOOTING PROCEDURES

Maintenance and troubleshooting measures are invoked as required, according to the outlines given in the manufacturers' manuals and utilizing the experience of the trained personnel who are responsible for the proper operation of the equipment.

ATTACHMENT I (Modified)

LOVE CANAL AIR MONITORING - QA/QC TARGET COMPOUNDS ON TENAX

(As per telephonic instructions from
Dr. Kenneth McGregor, GCA Technical Monitor)

COMPOUNDS TO BE QUANTIFIED:

1. Benzene
2. Carbon tetrachloride
3. Chlorobenzene
4. o-chlorotoluene
5. 1,2-dibromoethane
6. o-dichlorobenzene
7. 1,1,2,2-tetrachloroethylene
8. 1,2,4-trichlorobenzene
9. Toluene
10. Perfluorobenzene
11. Octafluorotoluene

COMPOUNDS TO BE QUALITATIVELY IDENTIFIED IF PRESENT:

1. Chloroform

3.0 SAMPLE COLLECTION

All samples except groundwater-related samples were collected by Geomet Technologies, Inc. Groundwater-related samples were collected by the supervisory Geologist subcontractor, JRB Associates, Inc., whose QA Plan is presented in Section 5.0 of this volume. The suggested QA Plan outline provided to Geomet Technologies, Inc. is given on the following page.

Subcontractor's
QA Project Plans
for
Multimedia Sampling at Love Canal

Outline

Section 1.0	Project Description and Organization
Section 2.0	Major Project Elements
Section 3.0	QA Objectives - State expected precision and accuracy to be achieved by each sampling technique, if possible to do so.
Section 4.0	Document Control Procedures - Overall framework, numbering system, sample tags, Custody Forms, Log Books, Data Sheets provided by GCA. State your internal operation of the system, identifying responsible person as Document Control Officer.
Section 5.0	Chain of Custody - Same as 4.0.
Section 6.0	Sampling Internal QC checks - Provide a table for each media sampled which lists operational checks for each sample collection technique. Control limits for each operational check should be provided with the corrective action to be taken if control limits are exceeded.
Section 7.0	Data Reporting Management System - Standardized data forms will be provided by GCA - both field data sheets and encoding forms for the data management system. Internal operation of the system, and data review before reporting should be described.
Section 8.0	Corrective Action Procedures - State how corrective actions will be implemented in the field - identify responsible personnel. The corrective actions should have been identified in Section 6.0.

Appendix A Sampling Procedures

Part 1. Sample Collection Procedures

Provided by GCA - includes field data sheets. Calibration of sampling equipment forms part of operating procedure - subcontractor provides for this equipment.

Part 2. Sampling Equipment Maintenance/Troubleshooting Procedures

GEOMET Technologies, Inc.
QA Plan

Table of Contents

Section		Pages	Revision	Date
1.1	Project Description	2	1	8-28
2.1	Project Organization and Responsibilities	4	1	8-28
3.1	Q.A. and Management Objectives	4	1	8-28
4.1	Sample Custody	18	2	9-09
5.1	Document Control and Revisions	4	1	8-25
6.1	Corrective Action and Feedback	6	1	8-27
7.1	Sampling Procedures	1	1	8-15
8.1	Documentation System for Instrumentation and Equipment	11	1	9-02
9.1	Quality Control Procedures for Instrument and Equipment	24	1	9-02
10.1	Performance of Internal Audits	5	1	9-02
11.1	Reports to Management	2	1	9-02

Distribution of Approved Q.A. Project Plan

Name	Document No.	Date
R. Elersick (GCA)	2 copies	9-3
J. Tuday (GTI)	1 copy	9-7
U. Perzan (GTI)	1 copy	9-3
J. Moran (GTI)	1 copy	9-7
B. Mason (GTI)	1 copy	9-7
Dr. Sibbett (GTI)	1 copy	9-8

1.1 PROJECT DESCRIPTION

Love Canal, located in Niagara Falls, New York, is the site of major environmental concern due to the chemical dumping in a defunct canal site over a period of decades. In order to help resolve this problem the EPA has requested assistance in a major environmental monitoring study which is required in order to determine the presence of hazardous chemicals in the environment and in the homes located in the vicinity of Love Canal.

GCA/Technology Division, currently under contract to the Environmental Monitoring and System Laboratory of the EPA, is the prime contractor responsible for managing the Love Canal Study. The program involves a multifaceted sampling and analytical effort designed to detect and quantify a variety of trace metals, volatile organics, pesticides, and other compounds in soil, sediment, air, biota, and water samples. GTI, a subcontractor to GCA, is responsible for conducting the sampling effort designed by GCA and the EPA. During the present study, GTI will provide environmental sampling and monitoring support necessary for making assessments of the health implication of any exposure identified. In this respect, GTI will be responsible for procuring and maintaining standardized sampling equipment, document the calibration and maintenance of all sampling equipment, maintain a spare parts inventory, conform to the chain-of-custody procedures developed by GCA, develop a Q.A. Project Plan which is consistent with the requirements of the sampling and monitoring plan, and provide the necessary expertise to assure that the sampling and monitoring conform to the protocol developed by GCA.

GTI recognizes its reponsibility in providing the support mentioned above and equally acknowledges that it is providing this support based on sampling procedures developed by GCA. GTI also recognizes that it is responsible to ensure validity of samples by complying to strict chain-of-custody procedures, however, it is also understood that matters concerning the security of homes during air sampling will be the sole responsibility of the prime contractor (GCA) and the EPA. As such, GTI will follow the security procedures developed and provided by GCA during the monitoring of all homes.

These security measures have been so designed by GCA as to guarantee the integrity of the sampling media and sampling equipment during periods of 12 to 24 hours. GTI in compliance to these security measures will document that the equipment has not been tampered with before, during, and after sample collection.

The sampling and monitoring network undertaken by GTI will include the collection of approximately 2,500 air samples from 79 sites using 177 air sampling trains and 13 HIVOL samplers; and over 750 samples of drinking water, sump water, surface water, soils, sediment, and biota. The results of this sampling effort will undergo sophisticated analysis by laboratories other than GTI under the overall management of the prime contractor (GCA). This sampling effort, of above-noted magnitude, is to be completed over a period of 3 months.

2.1 PROJECT ORGANIZATION AND RESPONSIBILITIES

All supervision of the environmental sampling/monitoring of air, water, soil, and biota; calibration of all instruments; and quality assurance will be performed by GTI personnel from the Gaithersburg, Maryland, Woodbury, New York, Rockville, Maryland, and Boulder, Colorado offices. Because of the unprecedented magnitude of this effort GTI will also use college and graduate students from the local college. These individuals will undergo extensive training and will be closely supervised by GTI employees who have been instructed in these particular tasks. The GTI management and staff personnel assigned to this project have extensive experience in all of the media for which sampling is required. In addition, both the Project Director and the Project Manager have been actively involved in major research programs requiring close cooperation with a number of governmental agencies.

The line of responsibility during this project is shown in Figure 2-1. Dr. Benjamin Mason, GTI Project Manager, serves as the key manager of all technical and administative activities in the field and will be responsible for coordinating and scheduling all project field effort. In addition, he will be responsible for interfacing with the GCA Project Officer and the onsite GTI Field Operation Manager John McWaters. The Quality Assurance Manager (Ugo Perzan) is responsible for ensuring the quality of environmental sampling and monitoring during this project. In this respect, he will provide the assurance that the quality assurance system is performing adequately by interfacing with the sample collectors to assure understanding of selection, collection, and handling practices. Achievement of a safe and healthy field operation is guided by the action of the Health and Safety Officer (John Pate). it is the responsibility of the Health and Safety Officer to provide a program that includes training, protective gear, and the constant insistence that field personnel be aware of and practice safe sampling procedures. From a Q.A. standpoint, accident prevention is vital. In general, maintaining quality performance depends on a smooth work flow. The sudden loss of an employee due to injury may upset the work flow sufficiently to compromise the quality of that day's sampling. The Field Operation Manager is responsible for coordinating all site sampling activities and the ordering of equipment and replacement parts, supply procurement, housing, transportation, etc. He has GTI purchasing authority.

The Field Operation Schedular has the responsibility of maintaining continuity to the sampling effort by providing the overall scheduling of the manpower requirement for each task proposed during this study. GTI's Field Operation Scheduler (James McFadden) is also responsible for and coordinating all sampling activities by developing the work plan necessary to comply with the scope of this project. The Document Control responsibilities have been assigned to several GTI staff members. Their major responsibility will include logging-in of all items determined to be control documents, ensuring that formal documentation of the operational procedures are updated, and that all project documents are accounted for by the close of the project. The GTI Calibration Monitor (Donald Cade) is responsible for assuring the required calibration and related record-keeping of all air monitoring instruments. The Calibration Manager is also responsible for the upkeep and trouble shooting of this equipment. The Task Managers for Air (Douglas Pelton), Water/Biota (James Brower), and soil (Benjamin Mason) are responsible for assuring the soundness of the technical approach used in the field for the sampling and monitoring of the different media. Task Managers are also responsible for reviewing the results of each days field effort and evaluate the integrity of the samples collected. The Field Coordinators for air (Harry Rector), Water/Biota (Robert Schreiber), and soil (Mike Milly) are experienced field persons who have participated in projects in a similar role. The Field Coordinators are present during all sampling activities and are responsible for assuring that proper field procedures are followed.

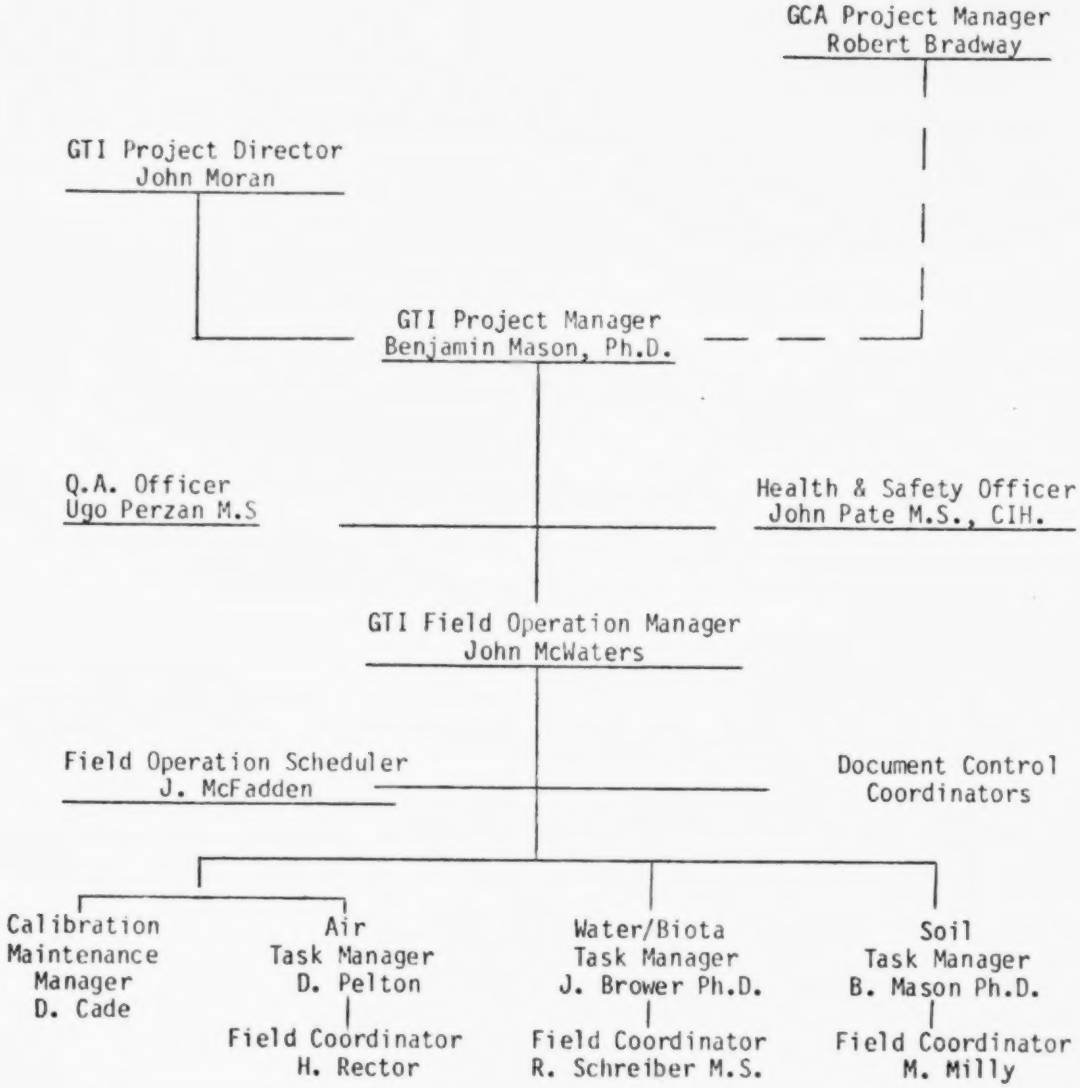

GCA Project Manager
Robert Bradway
GTI Project Director
John Moran
GTI Project Manager
Benjamin Mason, Ph.D.
Q.A. Officer
Ugo Perzan M.S
Health & Safety Officer
John Pate M.S., CIH.
GTI Field Operation Manager
John McWaters
Field Operation Scheduler
J. McFadden
Document Control
Coordinators
Calibration
Maintenance
Manager
D. Cade
Air
Task Manager
D. Pelton
Field Coordinator
H. Rector
Water/Biota
Task Manager
J. Brower Ph.D.
Field Coordinator
R. Schreiber M.S.
Soil
Task Manager
B. Mason Ph.D.
Field Coordinator
M. Milly

Figure 2-1. PROJECT ORGANIZATION

3.1 Q.A. AND MANAGEMENT OBJECTIVES

3.1.1 Q.A. Objectives

The objective of the Q.A. Project Plan is to provide concepts, direction, and methodology that can be used to maintain and improve the quality of data in the field investigations during this project. It is intended to provide direction needed for the development and implementation of quality control plans adapted to the data needs of the technical aspects of this project. Specifically, the Q.A. Project Plan is divided into three major activities:

Preventative - those activities that are conducted prior to sampling and that are intended to establish systems conducive to completeness and accuracy of data (e.q., preventative maintenance, calibration of instruments, development of a valid sampling plan, orientation and training of personnel).

Assessment of Precision - those activities that are conducted to measure mutual agreement among individual measurements of the same parameter, usually under prescribed similar conditions (e.g., replicate readings during calibration of instruments).

Corrective Action - those activities that are done, when error or possible error are detected, to correct the system (e.g., nonconformance and corrective action reports, recalibration of instruments, retraining of technical staff).

3.1.2 Oversight Role of Management

The achievement of quality objectives require quality people. The Task Managers are responsible for making quality assurance a personal objective of every employee working on this project. It is imperative that there be a mutual understanding of the nature of the quality assurance system and the reason for the various activities. Furthermore, it is managements duty to make sure that the personnel have the motivation, education, and training, to perform their jobs and to carry out the requirements of a quality assurance program.

GTI managers diligently strive to provide the proper atmosphere and conditions for motivating people to perform accurate work in the laboratory and in the field. Of course, this means providing a safe, efficiently designed facility in which personnel can work effectively and, at the same time, assuring the availability of supplies and equipment and an established system for accomplishing the work. In addition to cultivating this atmosphere, management at GTI maintains the philosophy that accuracy will not be sacrificed for expediency and has reassured staff members that they will not be reprimanded for reporting errors. These principals, although demanding, are essential for obtaining Q.A. objectives previously mentioned.

The achievement of quality objectives consists in managements insistence that proper training of all its employees be conducted as needed or required by a project. Training in this sense is defined as any process that increases the skills and abilities of the employee to do a specific job. Training is usually distinguished from education, which is the process of increasing one's understanding, obviously there are overlaps between the two processes. At GTI, recruitment of properly educated personnel is of primary importance to quality assurance, because understanding is essential for handling problems, or, for that matter, knowing that a problem exists. It is particularly important to train new employees properly, for this reason GTI's Task Managers must assume the sole responsibility for assuring that all personnel, old and new, know how to perform in a manner that produces the desired level of quality work. Since training is a mangement function that is closely related to quality assurance, all training programs need to be documented in the "Training Documentation" form (Q.A. Form No. 80-1). This form is vital in documenting the person giving the instruction, date, time, and location; in addition, the training objective(s) and the instrument/equipment involved during the training are also specified. The trainees need also to be listed with their signature acknowledging that they had attended the training session. To maintain consistency throughout the project, the objectives of the Q.A. plan and the training needs should be explicit enough to be compared with the objectives of the training course(s) under consideration.

Q/A Form No. 80-1

GEOMET Technologies, Inc.

TRAINING DOCUMENTATION

Instructor(s)	Affiliation	Title

Date:________ Time:________ Location:________

Training Objective(s):________

Instrumentation/Equipment Involved:________

Level of Training:

___ technicians ___ supervisory ___ part-time

Training Media Used:

___ lecture ___ film ___ slides ___ manuals ___ equipment Information bulletins

IF MANUALS OR OTHER PRINTED MATERIAL ARE PROVIDED, PLEASE ATTACH A COPY TO THIS FORM.

Trainees (name [print], title, signature)	Proficiency Verification Date	By
1)		
2)		
3)		
4)		
5)		

4.1 SAMPLE CUSTODY

Purpose: to assure integrity of the evidence obtained by providing permanent records of sample custody.

4.1.1 General Information

The sampling plan developed by the EPA for this study was designed to provide samples that have been collected in a fashion that will ensure the availability of quality data, therefore allowing for the enforcement action within the EPA and the Department of Justice. In view of this critical nature and the fact that the resultant data is expected to be used in court, GTI will conform with all chain-of-custody procedures to assure integrity of the evidence obtained by providing permanent records of sample custody. The procedures, forms, and sample tags used to provide sample custody have been developed by GCA with the assistance of the NEIC, and are addressed extensively in GCA's "Quality Assurance Project Plan." According to this plan, GCA will provide the appropriate chain-of-custody forms and tags; ensure the security of the homes being sampled and the equipment placed outside the homes; and provide QC-screened Tenax, polyurethane foam sampling modules, and preweighed HIVOL filters. GTI staff members, on the other hand, are responsible for initiating the chain-of-custody procedures when the samples are collected. In addition, GTI will comply with all security measures developed by GCA.

4.1.2 Chain-of-Custody Procedures

GTI in order to comply with the traceability of all samples will follow the chain-of-custody procedures listed below:

1. The field sampler is responsible for initiating chain-of-custody by signing his name in the appropriate box.
2. Sample is collected in accordance with standard sampling procedures found in the "Sampling Procedures Manual" provided by GCA.
3. If standard sampling procedures are not used, the field notebook is annotated and a written justification for deviation is maintained. Depending on the severity of the problem "Corrective Action System" proceedings may be initiated.
4. Upon completion of sampling, the sample is prepared in accordance with applicable sampling instructions (i.e., addition of preservative, refrigeration, proper sealing, etc.).
5. Chain-of-custody tags are prepared by the sampler by checking off the appropriate boxes, identifying the location of sample site, and sampler signature with the date and time of collection. Duplicates, field blanks, and sampler container numbers are recorded in the "Remark" box. When water samples are collected the "Remark" box of the sample tag will also include the following information: COC form number, sampling device used, depth of water, description of water flow and condition, pH, and chlorine concentration. If this information cannot all be entered in the "Remark" box it should be entered on the back of the tag.
6. Sample tag is attached or inserted to the immediate sample container in such a manner that it cannot be dislodged.
7. The chain-of-custody (COC) forms are completed by the originator/sampler. The originator fills in all requested information which is mostly retrieved from the sample tag (except in the case of air and water collection and external QC samples). For most sampling, more than one sample will be recorded on one form.

8. In the event of error in entering the appropriate information on the COC form, or the sample tag the originator should either void the whole form or tag, or void the line of entry which is found to be in error. In either event, changes or voided forms or tags should be initialed, dated, and retained.

9. At all times samples are maintained by the sampler in view or under limited access until custody is relinquished and formal documentation of shuch transer is completed. The samples should be handled by the fewest number of persons possible. In the event the sampler no longer has control of samples, he will relinquish it to the person now under control of same sample. When transferring custody, each person handling the sample should sign the form in the appropriate box and record the date and time of transfer. At no time should samples be left without being under control of a GTI staff person. When custody of the same group of samples changes hands several times, copies of the COC form is not required for each individual until it is relinquished to GCA Sample Bank. This is acceptable as long as the original custody record shows that each person who had received custody has properly relinquished it.

10. At the time when samples are being turned over to GCA's Sample Bank, the originator or the person that has received custody of the samples will be required to conduct an inventory of samples with GCA's sample custodian. This inventory is conducted by checking each sample against the chain-of-custody form, any discrepancies should be cleared up at this time. After this inventory, and only after this has been completed satisfactorily, the GTI person responsible for delivery sign his name in the top left "Relinquished by" box. The GCA person receiving custody will sign in the adjacent "Received by" box. The date/time should be the same for both signatures since custody is transerred at the same time. IN ALL CASES, IT MUST BE READILY SEEN THAT THE SAME PERSON RECEIVING CUSTODY HAS RELINQUISHED IT TO THE NEXT CUSTODIAN.

11. GCA Sample Bank will retain the original of the chain-of-custody form and give the copy to the initiator for GTI's record. All copies of this form are considered controlled documents which need to be retained in a central file at the GTI field office. The document control person assigned the task of logging in the I.D. number of each form and tag is responsible for doing so as soon as possible. At this time voided forms and tags are also recorded and retained until the time they will be returned to the GCA Sample Bank. All chain-of-custody forms and tags must be accounted for at the end of the study and, therefore, required to be placed in a secure place.

In addition to sample custody procedures listed above, the following items which may affect the integrity of samples need to be discussed:

4.1.2.1 Field Blanks

A field blank is prepared, handled, and analyzed in the same manner as normal carrying agents, except that it is not exposed to the material to be selectively captured. As such, the following procedure should be followed in initiating field blanks during the present study:

1. One field blank for each sample type (e.g., volatile, semivolatile, pesticide, etc.), for each sampling team, and for each collection media (air, water, soil, sediment) to be included with the regular samples on each sampling day.
2. During air sampling three media are used (Tenax, polyurethane foam, and air filters). Field blanks for each media should be carried out to one of the sample sites but should never be removed from their sample container. Each team is required to carry the appropriate field blanks during one days sampling effort. Field blanks should be given a sample tag for identification and recorded on the chain-of-custody form as described in previous section. Field blanks need to be distinguished from other samples by recording "Field Blank" in the "Remark" box and relinquished to the GCA Sample Bank with the rest of the samples.
3. During the water sampling (i.e., sumps, drinking water, sewer and drain water, and surface water) a sampling container filled with reagent water is to be used as a field blank. These field blanks are to be carried out to the field and returned to the Sample Bank as described under air sampling.
4. During sediment sampling (i.e., surface water, sewer and drain, and sumps) sample containers filled with reagent water will constitute field blanks. These field blanks are to be carried out into the field and returned to the Sample Bank as described in the air sampling.
5. During soil sampling field blanks will be the same as those described in the sediment sampling.

4.1.2.2 Replicates

During this study, replicates are defined as repeated collections of the same sample, by the same collector, at essentially the same time and under the same conditions. Replicates may be performed to any degree, e.g., duplicates, triplicates, and so forth. Replication of samples for each type of analysis and sample type is determined by the "Sampling Procedures Manual". As with the field blanks, replicates should be properly identified with the appropriate sample tags. The "Remark" box on each sample tag should also signify replication of sample by recording 1 of 2, 2 of 2, and so on. Similarly, this information should be recorded on the chain-of-custody form. On occasions where extra samples are requested by either the EPA or GCA, the appropriate information should be recorded on COC forms and in the field notebook. All replicate samples are to be returned to the Sample Bank with other samples as prescribed by the chain-of-custody procedures.

4.1.3 Transmission of Information

4.1.3.1 Field Notebooks

Entries into notebooks is the collection of information relevant fo some unit topic of the study. Request for information not recorded on the sample tag, chain-of-custody form, and data sheet almost invariably arise the need to add information to a notebook.

Field notebooks are provided by GCA's Sample Bank and are serialized by GTI staff members according to GCA specifications. A record of each notebook is maintained in a master log book at the GTI field office. These notebooks are assigned to individuals who are involved in sampling, or to a specific home that is being used during the air monitoring portion of this study. Field notebooks are control documents which contain information that may be useful during the interpretation of data, therefore entries should be clear and legible with minimum crossing out.

4.1.3.2 Flow of Information

In theory, the flow of information is regulated by a control system; this system is the written documentation that describes how each process works and what steps are required (e.g., methods used for sample collection, information on handling and storage of samples, instructions on operation, instrument maintenance, safety rules, etc.). However, GTI realizes that because of the magnitude and diversity of the present study, the need may arise for the modification of certain study procedures. It is during such modifications that the flow of information becomes critical to the outcome of the study. Transmission of this type of information first requires some form of verification. At GTI, this verification is obtained from the Project Manager. No modification to any procedures will be initiated by GTI staff members unless accompanied by written documentation of such approval. It is understood that before modifications or changes are announced, the GTI Project Manager will have received approval or derective from both GCA's Project Manager and an EPA representative.

The GTI Project Manager is solely responsible for informing his staff of any changes or modification in either the scope of work or procedures. Staff members will receive communication in a memo format. This communication will identify the specific procedure that is being changed or modified, and will clearly explain how to implement these changes. Changes or modifications affecting this manual will be entered as revisions to the manual and dated.

Any communication by telephone will require that a full duplex transmission system be implemented. Such a system requires that the receiver of the message repeat back to the sender the original message to check for accuracy.

4.1.4 Sample Identification

Accurate sample identification is an essential part of sample custody. The sample must be positively identified at the time of collection to the time of analysis. During this study sample identification is achieved by three basic steps:

1. Sample containers are given individual numbers after they have been prepared according to specified procedures found in the "Sampling Procedure Manual". Basically, preparation of sample containers require two types of washing. Containers receiving samples to be tested for volatile organics and inorganics do not receive methylene chlorine washing while containers receiving samples for other analysis are washed with methylene chloride. It is suggested that the person responsible for the preparation of containers develop a method or code for distinguishing between the two types of washing. All containers receiving an identification number are assumed to be clean, and recorded in a logbook for cross reference.

2. Sample identification tags are provided by the GCA Sample Bank for air, water, soil, sediment, and biota (see Figures 4-1 and 4-2). These tags are a different color for each media and are serially numbered starting with different blocks of numbers for each media. Sample tags are attached to the collected sample and the container number is recorded on the "Remark" box of the tag. This procedure provides an extra measure for the identification of sample in case a tag becomes dislodged from the sample container.

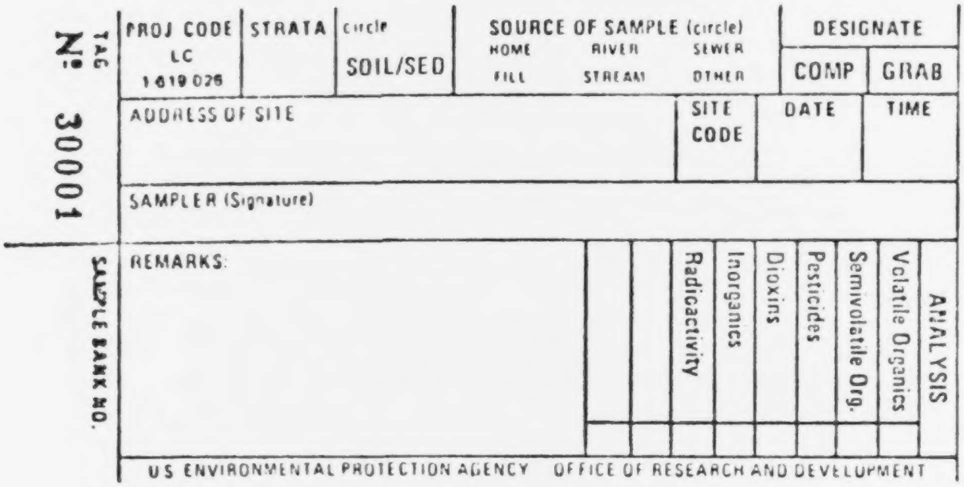
3. Chain-of-custody forms are an additional measure to record the proper identification of samples. These forms are two part carbonless copy form and are also provided by GCA Sample Bank (example of three of these forms are found in Figures 4-3, 4-4, and 4-5). The information that needs to be recorded on these forms closely correspond to the information found on the sample tags. Specifically, the tag number transcribed on the left hand side of the form, the container number recorded in the "Remarks" column on the right hand side, and sample type all act as confirmation of sample identification.

4.1.5 Procedures to Ensure Validity of Sample During Air Monitoring

In order to establish that a sample is valid, it is also necessary to document that no tampering has occurred either to the sample itself, or the sampling equipment. Under the direction of GCA, this is done by the use of evidence tape, locks, and documented entries in field notebooks. Field notebook and custody record entries will document any tampering that may have occurred; the absence of any such comments or entries indicates no tampering during the monitoring period.

4.1.5.1 Security During Home Ventilation

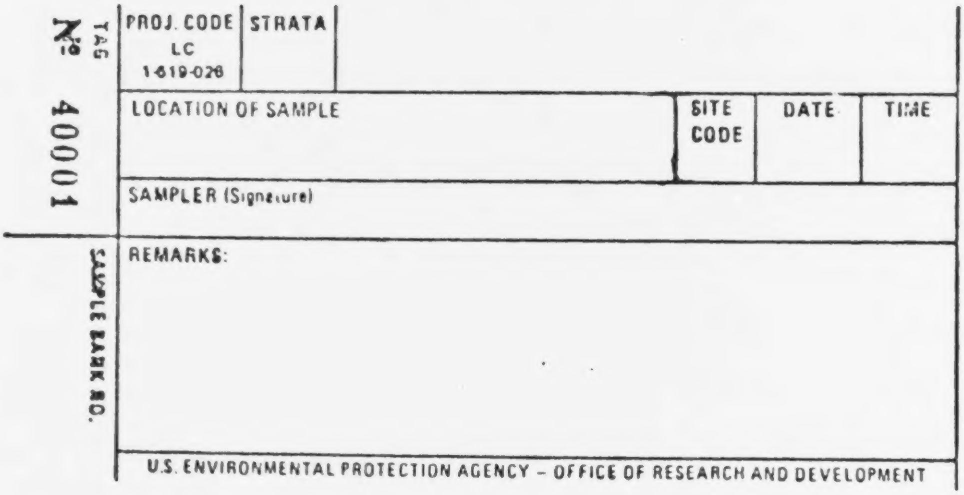
The following procedures are to be followed when ventilating homes before air monitoring:



TAG Nº 30001

PROJ CODE LC 1-619-026
STRATA
circle SOIL/SED
SOURCE OF SAMPLE (circle) HOME RIVER SEWER FILL STREAM OTHER
DESIGNATE COMP GRAB
ADDRESS OF SITE
SITE CODE
DATE
TIME
SAMPLER (Signature)
REMARKS:
ANALYSIS
Volatile Organics
Semivolatile Org.
Pesticides
Dioxins
Inorganics
Radioactivity
SAMPLE BANK NO.
U.S. ENVIRONMENTAL PROTECTION AGENCY OFFICE OF RESEARCH AND DEVELOPMENT

SOIL/SEDIMENT SAMPLE TAG

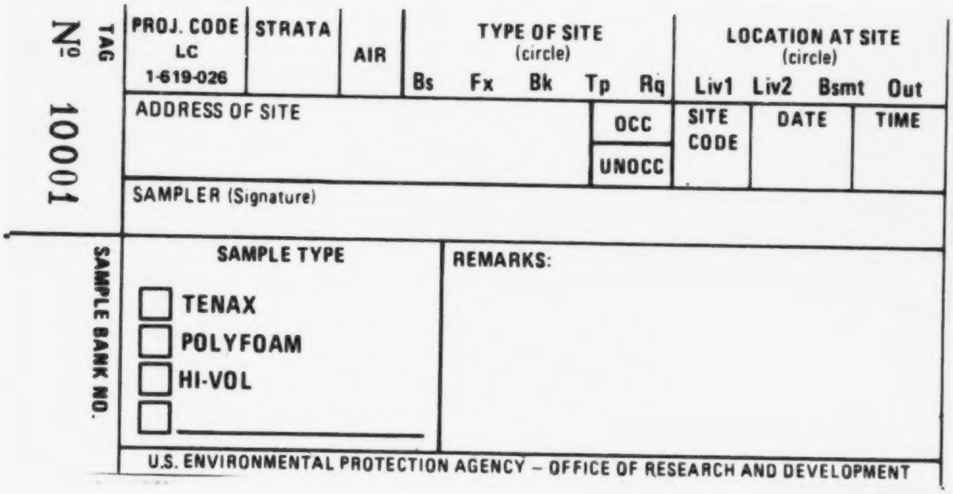


TAG Nº 40001

PROJ. CODE LC 1-619-026
STRATA
LOCATION OF SAMPLE
SITE CODE
DATE
TIME
SAMPLER (Signature)
REMARKS:
SAMPLE BANK NO.
U.S. ENVIRONMENTAL PROTECTION AGENCY – OFFICE OF RESEARCH AND DEVELOPMENT

BIOTA/OTHER SAMPLE TAG

Figure 4-1



TAG Nº 10001	PROJ. CODE LC 1-619-026	STRATA	AIR	TYPE OF SITE (circle) Bs Fx Bk Tp Rq	LOCATION AT SITE (circle) Liv1 Liv2 Bsmt Out

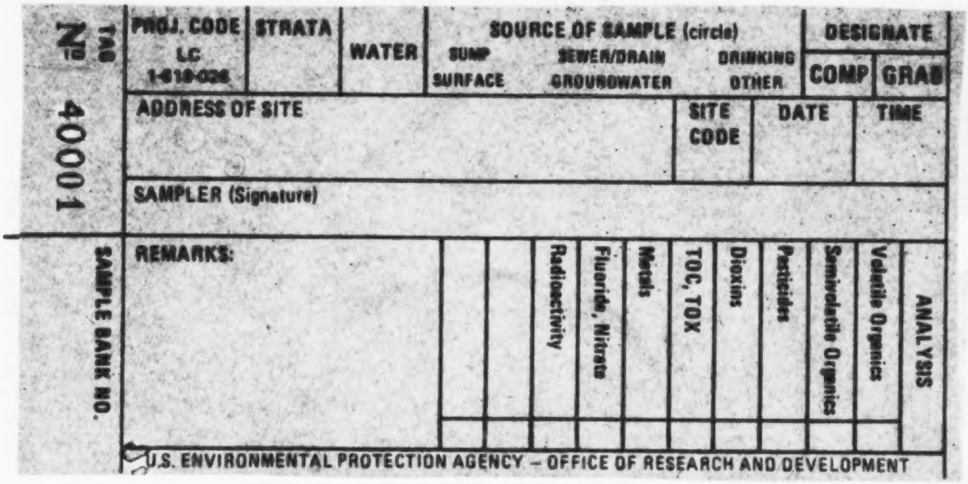
ADDRESS OF SITE	OCC UNOCC	SITE CODE	DATE	TIME

SAMPLER (Signature)

SAMPLE BANK NO.	SAMPLE TYPE ☐ TENAX ☐ POLYFOAM ☐ HI-VOL ☐ ______	REMARKS:

U.S. ENVIRONMENTAL PROTECTION AGENCY – OFFICE OF RESEARCH AND DEVELOPMENT

AIR SAMPLE TAG



TAG Nº 40001	PROJ. CODE LC 1-619-026	STRATA	WATER	SOURCE OF SAMPLE (circle) SUMP SEWER/DRAIN DRINKING SURFACE GROUNDWATER OTHER	DESIGNATE COMP GRAB

ADDRESS OF SITE	SITE CODE	DATE	TIME

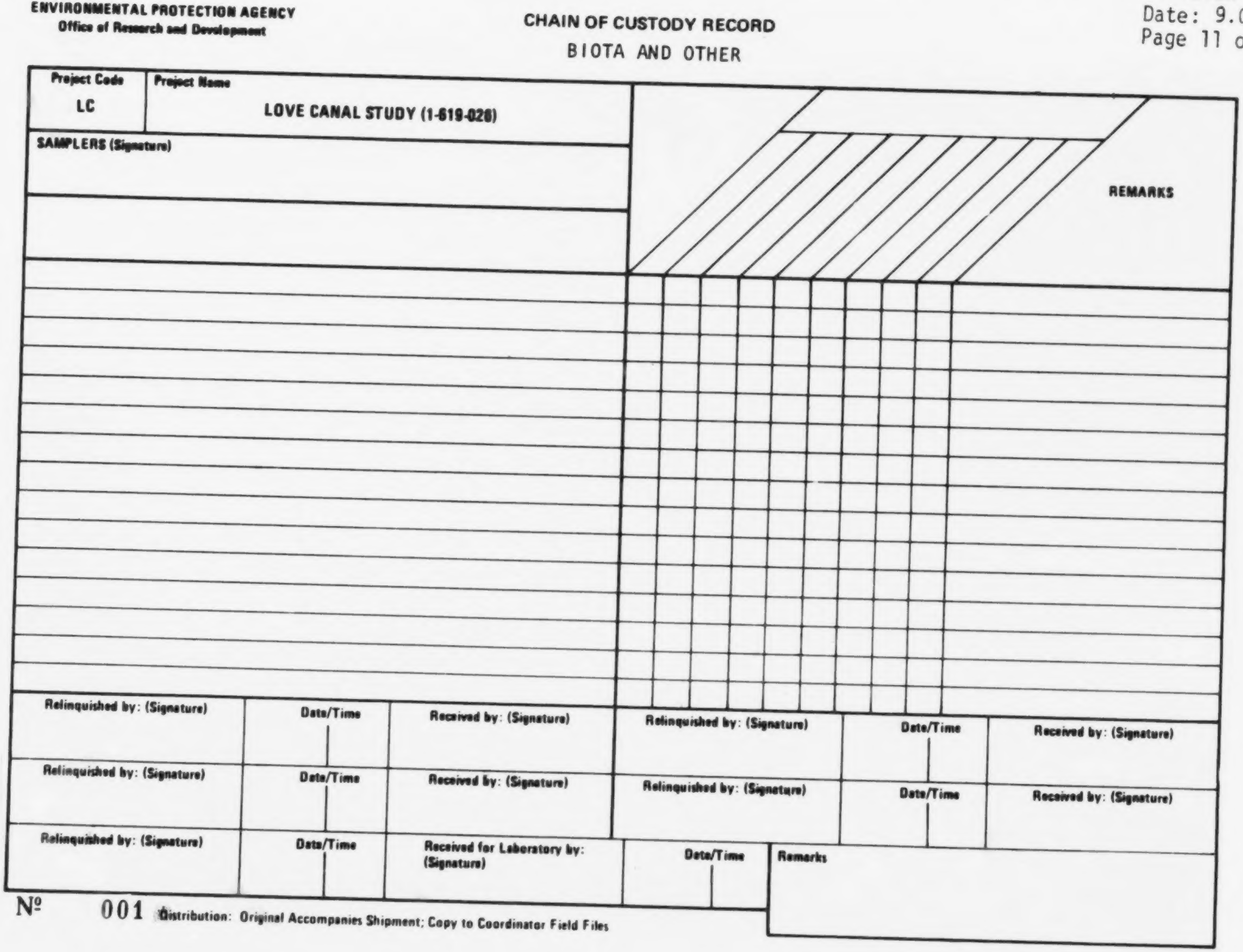
SAMPLER (Signature)

SAMPLE BANK NO.	REMARKS:			Radioactivity	Fluoride, Nitrate	Metals	TOC, TOX	Dioxins	Pesticides	Semivolatile Organics	Volatile Organics	ANALYSIS

U.S. ENVIRONMENTAL PROTECTION AGENCY – OFFICE OF RESEARCH AND DEVELOPMENT

WATER SAMPLE TAG

Figure 4-2



ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development

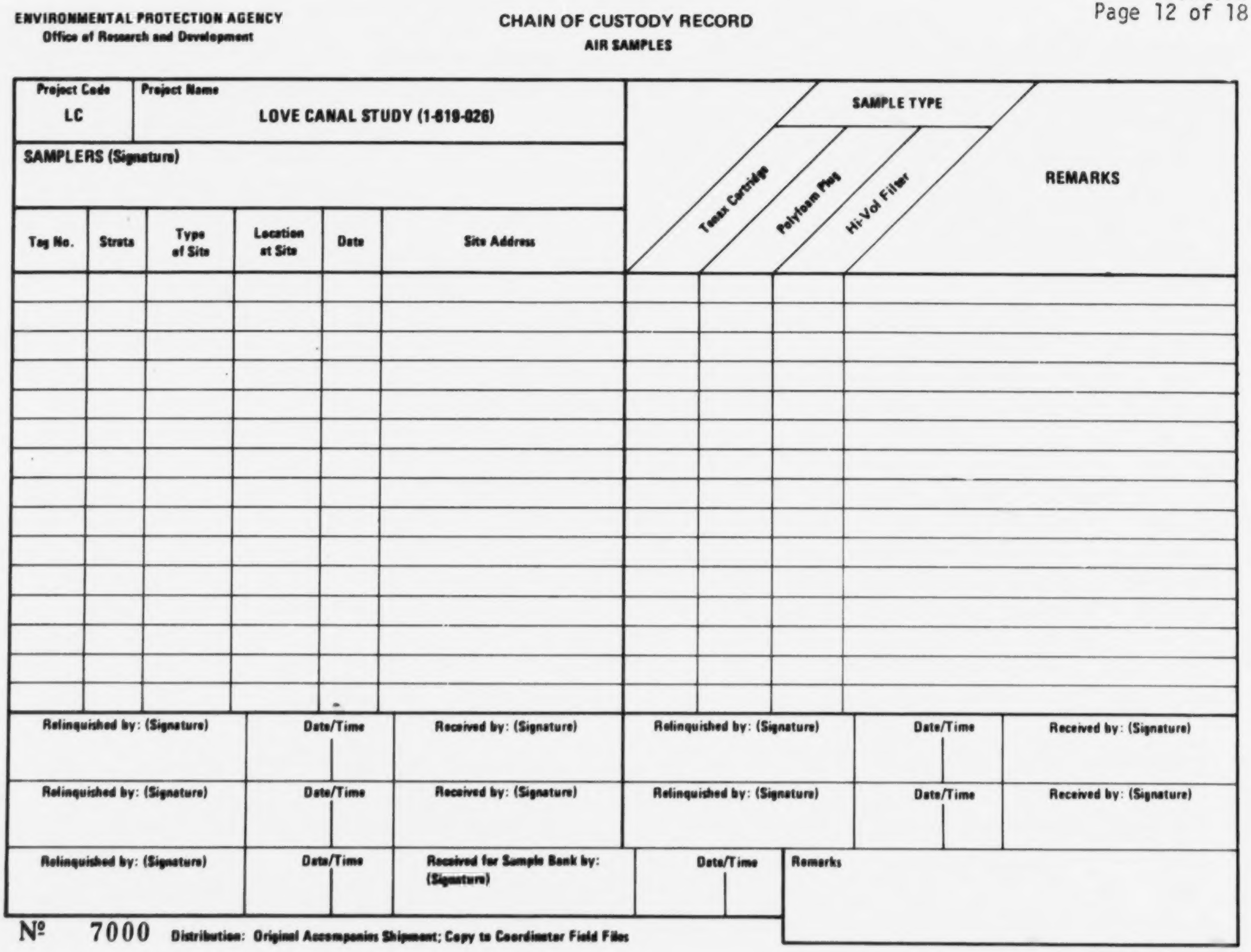
CHAIN OF CUSTODY RECORD
BIOTA AND OTHER

Project Code LC	**Project Name** LOVE CANAL STUDY (1-619-020)	**REMARKS**
SAMPLERS (Signature)		

Relinquished by: (Signature)	**Date/Time**	**Received by: (Signature)**	**Relinquished by: (Signature)**	**Date/Time**	**Received by: (Signature)**
Relinquished by: (Signature)	**Date/Time**	**Received by: (Signature)**	**Relinquished by: (Signature)**	**Date/Time**	**Received by: (Signature)**
Relinquished by: (Signature)	**Date/Time**	**Received for Laboratory by: (Signature)**	**Date/Time**	**Remarks**	

№ 001 **Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files**

Figure 4-3



ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development

CHAIN OF CUSTODY RECORD
AIR SAMPLES

Project Code	Project Name
LC	LOVE CANAL STUDY (1-619-026)

SAMPLERS (Signature)

Tag No.	Strata	Type of Site	Location at Site	Date	Site Address	SAMPLE TYPE Tenax Cartridge	 Polyfoam Plug	 Hi-Vol Filter	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

Nº 7000 Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

Figure 4-4

ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development

CHAIN OF CUSTODY RECORD
WATER/SOIL/SEDIMENT

Project Code LC

Project Name LOVE CANAL STUDY (1-619-026)

SAMPLERS (Signature)

										ANALYSIS								
Tag No.	Strata	Water	Soil	Sed.	Source of Sample	Comp.	Grab	Date	Site Address	Volatile Organics	Semivolatile Organics	Pesticides	Dioxins	TOC, TOX	Metals	Fluoride, Nitrate	Radioactivity	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Sample Bank by: (Signature)	Date/Time	Remarks	

№ 4000 Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

Figure 4-5

1. Pick up keys to homes from the GCA field office.

2. Drive to first house, record the address, time, team members names, and other comments in the field book.

3. Carry equipment to door. DO NOT WEAR RESPIRATORS AT THIS TIME. Unlock door and enter. NOTE: If keys do not unlock door, go to caretaker and get master key, and then get in touch with GCA field office. Record in field notebook any problems with entry.

4. Once entry is made and door closed behind team, all personnel should adorn their respirators.

5. Monitor home with Century OVA 98A and Victoreen geiger counter.

6. Health and Safety personnel should open door to basement and lead team down to basement.

7. Basement area is monitored with OVA geiger counter and readings recorded in field notebook. Particular attention and safety precautions should be paid to the sumps.

8a. If readings are below 10 ppm or 10 mr, respirators are removed and ventilation proceeds normally.

8b. If all readings are between 10 and 100 ppm or 10-100 mr ventilation operation proceeds with respirators being worn at all times.

8c. If readings of house are greater than 100 ppm or mr, the chief health and safety officer and the GCA field office are notified immediately and ventilation operation is terminated until further direction from chief health and safety officer is obtained.

9. The house (including closets, drawers, etc.) are searched for containers of organic materials. If any are found, they are placed in a plastic bag, tagged with house identification, time and signature. Each item should be listed on duplicate memo paper, and the original enclosed in the bag and returned to GCA field office. All items should be returned to the house at the end of the study.

10. Check if house has an operational heater with fan. If fan is not operational notify maintenance person (Howard Calvin, 283-1611).

11. Remove plywood from two windows on second floor, open windows and install fan in upwind window space.

12. Place another fan at top of cellar stairs (door should be opened).

13. Start fans including house fan (if available) and record the time in field notebooks.

14. Ventilate for two (2) hours.

15. Turn off fans, recording the time in the field logbook, and move basement fan to second window on the second floor.

16. Turn on fans, recording the time and ventilate for two hours.

17. Turn off fans, record time, remove fans, and replace plywood on windows.

18. Place tripods at inside sample points (basement and living area).

19. Place outside tripod and HIVOL sampler(s) inside house.

20. Use security tape on all inside windows and doors except for front door.

21. Turn off all lights, lock and seal front door.

22. Record in field notebook the time of departure, team members, and remark on the security procedures taken to secure house.

4.1.5.2 Security During Monitoring of Homes

After homes have been properly ventilated and secured, the following procedures will be followed and documented in the field notebook:

1. At arrival at the house to be monitored the field team will survey the outside of the house and record in the field notebook (each home to be sampled will be assigned a logged notebook) the date, time, and names of field team.

2a. Examine the front door for possible signs of tampering, document that evidence tape is still securely afixed to the front door. Proceed to unlock the door and enter home.

2b. If tampering is evident (i.e., evidence tape is ripped) document the situation including a judgement as to whether tampering was involved. Do not proceed with sampling, contact GCA onsite Q.A. coordinator or EPA representative.

2c. If GCA or EPA personnel are not available proceed to next house and have GTI field office follow up on contacting the appropriate GCA or EPA personnel to determine if sampling should proceed at that home for that day.

3. Enter house that has not been tampered with and proceed to document condition of inside taped areas. If all tape is unripped, proceed with sampling. If tape is ripped proceed with steps 2b and 2c.

4. Set up monitoring equipment in the basement and living area as specified by "Sampling Procedures Manual".

5. Proceed to set up sampling equipment outside of house. Make sure that appropriate security for equipment is provided by GCA.

6. When leaving house even for a short time, tape the entry door on the outside, and record departure and re-entry in notebook.

7. When all equipment has been properly set up, go through operational checks to ascertain that all equipment is working. If any of the equipment is malfunctioning contact GTI field office and proceed to next house.

8. Leave house, place strip of evidence tape on outside of sampling team entry door, and record the time of departure in notebook.

9. At the end of monitoring period (12 hours for volatile and pesticide) return to house, and follow steps 1 through 3.

10. Remove samples and sampling equipment and determine if equipment has been operating properly.

11. Proceed with step 8.

4.1.5.3 Security of Sampling Equipment

It is imperative to demonstrate that all equipment used during this study has not been tampered with before, during, and after sample collection. During sampling most equipment is under the control of the sampling team and, therefore, are assumed to be secured. However, during the air monitoring of homes and meteorological monitoring, the equipment is not in control of sampling team for an extended period of time. As such, certain security measures have been instituted to protect all equipment. These measures were developed by GCA to protect homes and the equipment during air monitoring and have been discussed in previous sections. If equipment is in a house that has not been tampered with during collection, it may be assumed that tampering of equipment has not occurred.

Meteorological equipment found in GTI's mobile trailer will also be secured by taking similar measures. Specifically, the mobile trailer found in strata 11 will have limited access, since it is found within a fenced in area secured by the EPA. In addition, all windows and exit door of the trailer will be secured with security tape. Furthermore, if possible, equipment in the trailer may also be secured with custody seals. Do not use tape on samplers collecting volatile or semivolatile organic samples because of possible contamination from plastics or adhesive.

As often as possible, GTI personnel will make onsite visits to determine if meteorological equipment is operational, during these visits they will also note if any tampering with the equipment has occurred. Any signs of tampering will be noted in field notebooks and reported to GCA field office.

Security of equipment during storage at GTI field office (i.e., before and after sampling) is accomplished by keeping the equipment under lock and key and placing strips of evidence tape on storage room doors. Access to this storage area will only be allowed to GTI staff members. In addition, accountability of all sampling equipment will be accomplished by logging in and out during sampling. This measure will allow for the tracking of all equipment during sampling and when equipment is out for repairs. In the event that tampering or forced entry into the storage area has been determined all equipment will be checked, accounted for, and recalibrated.

5.1 DOCUMENT CONTROL AND REVISION

Purpose: to ensure that all project documents are properly accounted for and to provide a system for updating formal documentations of operating procedures.

5.1.1 Document Control Description

GTI is solely accountable for all documents properly assigned to it by either GCA or the EPA, and for all internal documents prepared during this study. Document control at GTI is attained by assigning responsibilities for the issuance, control, and maintenance of documents to specific staff members. Table 5-1 lists the principal items subject to document control and the persons responsible for recording and maintaining these items. Items identified as controlled documents are promtly logged-in in a bound book with prenumbered pages in order to retain a permanent record of the items disposition. At the conclusion of GTI's subcontract activity, all controlled documents with records will be provided to GCA for inclusion in an overall project document inventory.

5.1.2 Project Code

The assigned project code for the Love Canal Study is LC 1-619-026. All reference to this project will include the project code and, therefore, will appear on sample identification tags, chain-of-custody forms, logbooks, data sheets, control charts, project memos, document control logs, corrective action forms and logs, Q.A. Project Plan, and other project related records. The Project Code should not be confused with GTI's internal coding of Q.C. forms and equipment identity assignments (Section 8.1). These numbers are assigned for the purpose of traceability of internal forms and equipment and do not require the Project Code for identification.

5.1.3 Revision Format for Q.A. Project Plan

All revisions to GTI's Q.A. Project Plan will conform to the format recommended by EPA's "Quality Assurance Handbook for Air Pollution Measurement Systems" (EPA-600/9-76-005). According to this format the following information will be included at the top of each page:

Section No.:
Revision No.:
Date:
Page:

The "Section No." at the top of each page identifies major two-digit sections, where applicable. "Revision No." represents the most current version of the section in question, where the first version is represented as "0". "Date" represents the date of the latest revision. "Page No." includes not only the number of the specific page, but also the total number of pages in the section. For each two-digit level, the text begins on a new page. This format allows for the grouping of pages, therefore facilitating revisions of the section in question. When a section is expanded the number of the preceeding or original page is included in the new page, and a letter is added. For example, if page 2 of 6 was revised and expanded to include an extra paragraph, the overflow would appear on a page designated 2a. The original page 2 would then be removed from the text and replaced by revised page 2 and 2a. In the event that a whole section is revised the enumeration of pages may preclude reference to previous page numbers. Upon revisions, the Table of Contents which follows the same structure as the text will also be revised by retyping, or striking out the old revision number and printing the current revision number with the date the revision was made.

5.1.4 Q.A. Project Plan Distribution Record

The "Distribution of Approved Q.A. Project Plan" follows the Title Page and is a permanent record of all individuals receiving a copy of the Q.A. Project Plan. This listing is updated to include all persons receiving a copy of the document, therefore allowing for the immediate distribution of all revisions. Changes to the Q.A. Project Plan may be made by the issuance of (1) an entirely new document (2) an entirely new section (3) replacement pages or, (4) an errata sheet or by pen and ink posting on the original document. The recipient of these changes will be required to remove and destroy all revised pages, sections, etc. from his or her copy and/or make pen and ink correction as noted.

Unless otherwise instructed, all copies of the Q.A. Project Plan will be accounted for upon termination of this project.

Table 5-1

Controlled Documents Maintained by GTI

Item	Point of Origin	I.D. # or Enumeration	Person(s) Responsible	Recorded in Log Book Yes/No
Chain of Custody Form	GCA Sample Bank	Preserialized	J. McFadden	Yes
Sample Collection Tags	GCA Sample Bank	Preserialized	J. McFadden	Yes
Field Notebooks	GCA Sample Bank	LC 1-619-026-__	J. McFadden	Yes
Air Monitoring & Home Security Notebooks	GCA Sample Bank	LC 1-619-026-__	J. McFadden	Yes
Photos, Maps, & Drawings	GCA/GTI	Identified by originator signed & dated	J. McFadden & Task Managers	Yes
External Communications	Various people	By originator & date	Recipient	No
Sampling Procedure	GCA Q.A. Manager	None	GT U. Perzan B. Mason	Yes
Q.A. Memos	Q.A. Staff	By originator & date	U. Perzan	Yes
Project Work Plan	GTI	None	J. McFadden	No
Q.A. Project Plan	GTI	Numbered in series as assigned	U. Perzan	Yes

6.1 CORRECTIVE ACTION AND FEEDBACK

Purpose: to detect out-of-control or unsatisfactory conditions which may affect the quality of work, and to ensure a systematic and timely mechanism for reporting to those who can correct them.

6.1.1 Types of Corrective Actions

Two types of corrective actions can be implemented once the problem has been determined. Malfunctioning or failure of monitoring equipment during the sampling period requires immediate corrective action or on-the-spot remedial action. Application of on-the-spot corrective action can be effected immediately by operators since established corrective operational procedures are delineated in the "Instrument and Equipment Quality Control Procedure" (Q.C. Form 80-4). Calibration, operational checks and maintenance procedures have been developed to detect instances in which corrective action is necessary. If needed, trouble-shooting guides for field operators are generally found in instrument manufacturer's manuals. On-the-spot corrective actions routinely made by field technicians will be recorded as normal operating procedures, and no specific action other than notations in operations log books need be initiated.

For major problems, i.e., difficulties encountered during field operation which may require changes in procedures, or in situations where practices are demonstrated to be ineffective in obtaining the required results, therefore resulting in questionable data or samples, a formal system of reporting and recording corrective action needs to be initiated. This system, to be effective, depends on the cooperation of all staff members who are motivated in performing their jobs and carrying out the requirements of a quality assurance program. Concurrently, management must assure its staff members that accuracy will not be sacrificed for expediency and that they will not be reprimanded for reporting any inconsistencies in the sampling procedures.

6.1.2 Implementation of Corrective Action Systems

Figure 6-1 demonstrates a flow diagram of the Corrective Action System to be used during the Love Canal Study. This system is basically dependent on the implementation of the following steps:

1) Define the problem
2) Assign responsibility for investigating it
3) Investigate and determine the cause of the problem
4) Determine a corrective action to eliminate it
5) Assign or accept responsibility for implementing the corrective action
6) Establish effectivity and implement the correction.
7) Verify the corrective action has eliminated the problem.

The primary test of the Corrective Action system is the corrective-action request form (Figure 6-2). This form is preserialized and available at the GTI field office for any staff member who has become aware of either an inconsistency or failure of a particular procedure to produce the required data. For example, the inefficiency of a particular field method in recovering a particular type of sample may be sighted by the field operator.

The operator by completing a corrective action request form would be the originator of a corrective action and would be responsible in following the action through until it becomes resolved. As the originator he would state the nature of the problem, the apparent cause and during what phase of the operation the problem was identified. He would then assign the responsibility for replying to his action to an individual most able to investigate and/or solve the problem. The person receiving the request for corrective action (usually the Task Manager) would either determine that the action required is beyond the scope of his position, and forward it to the Project Manager, or would proceed to spell out the corrective action planned. He would also indicate if this was a temporary or permanent action, and forward it to the project manager for approval. Once approved, the person replying to the corrective action is responsible in selecting the person or persons for implementing the plan. The originator is also responsible for logging in the corrective action request form number on the corrective action master log (Figure 6-3) and to follow through the corrective action until the final close-out date.

If the problem is reported by letter, by memo, or by other means, the recipient should complete a covering form. All of the corrective action request forms and master logs are maintained in a central file for management visability. During Q.A. audit, these files will be examined to determine: a) the type of problems that have occurred during the sampling period; b) establish the effectivity of the corrective action implemented and; c) to verify that the corrective action has eliminated the problem.

FIGURE 6.1 GTI'S CORRECTIVE-ACTION FLOW DIAGRAM

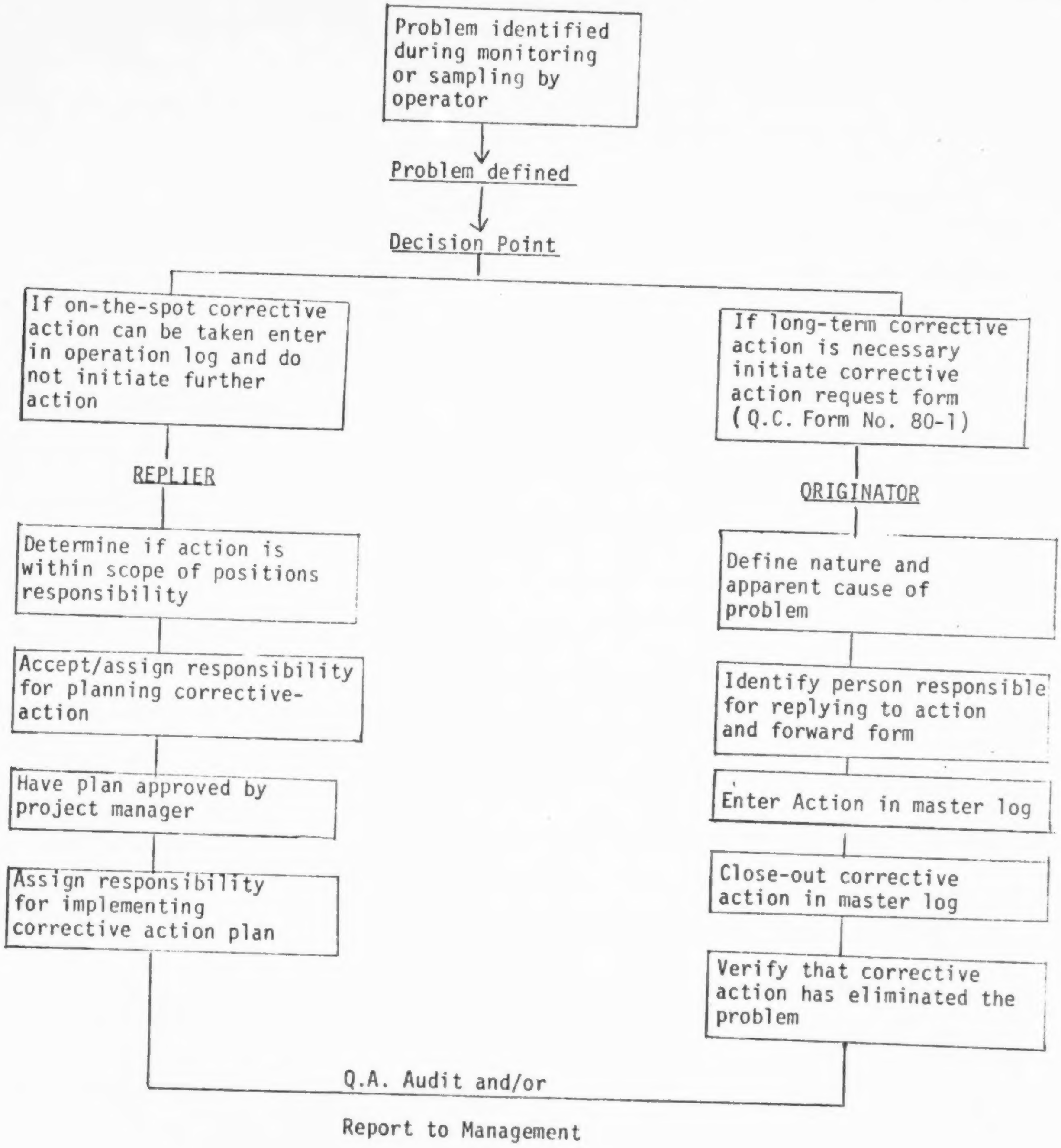

Problem identified during monitoring or sampling by operator
Problem defined
Decision Point
If on-the-spot corrective action can be taken enter in operation log and do not initiate further action
If long-term corrective action is necessary initiate corrective action request form (Q.C. Form No. 80-1)
REPLIER
ORIGINATOR
Determine if action is within scope of positions responsibility
Define nature and apparent cause of problem
Accept/assign responsibility for planning corrective-action
Identify person responsible for replying to action and forward form
Have plan approved by project manager
Enter Action in master log
Assign responsibility for implementing corrective action plan
Close-out corrective action in master log
Verify that corrective action has eliminated the problem
Q.A. Audit and/or
Report to Management

Figure 6.2
GTI Corrective Action Request Form
Q.C. Form No. LC 80-1

Serial No.__________

*NOTE: This is part of a corrective action system, and the originator is responsible in following the request through to resolution. Responsibility for a reply should be assigned to an individual most able to investigate and/or solve the problem.

Originator: ____________________ Date: ____________________

Person responsible
for replying: ____________________ Title: ____________________

PROBLEM IDENTIFICATION (to be completed by originator)

A) State nature of problem: ____________________

B) State apparent cause of problem: ____________________

C) During what phase of operation was the problem identified? ____________________

Signature: ____________________

Form adapted from EPA Air QA Handbook (EPA-600/4-77-027a) 1977.

Appendix Q
Plan 3.1

*NOTE: Please return this form to the originator within 2 days of receipt with the corrective action planned. If you determine that the action required is beyond the scope of your position, please indicate below who is responsible.

Name: ______________________ Title: ______________________

Corrective Action Planned: ______________________

____

____

____

____

____

Is this a temporary action? YES____ NO____

Is this a permanent action? YES____ NO____

Signature: ______________________ Date: ______________________

Plan approved by: ______________________ Date: ______________________

Persons responsible for implementation of plan:

______________________ Title: ______________________

______________________ Title: ______________________

Follow up action required? YES____ NO____

DISTRIBUTION

Original: to Originator

Second Copy: to Implementor of Corrective Plan

*NOTE: Originator Log in Corrective Action in "Corrective Action Master Log."

Figure 6.3

GEOMET TECHNOLOGIES, INC.
CORRECTIVE ACTION MASTER LOG
Q.C. FORM NO. 80-6

Corrective Action Request Form No.					
Date Submitted					
Submitted By					
Assigned To					
General Problem Description					
Corrective Action Responsibility					
Corrective Action Taken					
Demonstration of Effectiveness					
Final Close-out Date					

Form adapted from EPA Air Q.A. Handbook (EPA-600/4-77-027a)

7.1 SAMPLING PROCEDURES

Sampling procedures for this study has been provided by GCA in their "Quality Assurance Project Plan for Multimedia Monitoring at Love Canal" Appendix A Sampling Procedures.

8.1 DOCUMENTATION SYSTEM FOR INSTRUMENTATION AND EQUIPMENT

8.1.1 Introduction

GTI's quality control program for management of instrumentation and equipment is described in this section. The program encompasses measures for control of all items from date of receipt through routine use in the field office. Systematic documentation is provided for summarizing calibration, operational check, and maintenance requirements and frequencies. Specific details for these protocols and intervals are discussed in Section 9.1.

8.1.2 Procurement Control

8.1.2.1 Inspection

Inspection upon receipt constitutes the initial step in the chain of quality control activities involving instrumentation and equipment. Documentation is provided through use of the Shipment Receipt Record (QC Form No. 80-2) shown in Figures 8-1 and 8-2. The form includes provisions for recording the incidence of damage, missing or erroneous items, and serial or identity numbers. An appropriate signoff block is provided for the individual executing the inspection.

8.1.2.2 Identity Assignments

GTI realizes that unique identification for instrumentation and equipment items is necessary for tracking of performance and assignment to

Q/C Form No. 80-2
Page 1 of 2

GEOMET Technologies, Inc.

SHIPMENT RECEIPT RECORD
(Instrumentation and Equipment)

Item: ____

Manufacturer: ______________________ Model No.: __________

Vendor/Supplier: ______________________ P.O. No.: __________

Inspector: Name (print) ________________ Title ________________

Signature ________________

Date of Receipt: __________ Number of Items: ________________

Manufacturers Serial No. (if sequential, list here): __________ to __________
(if not sequential, list individually on reverse side)

If Manufacturers Serial No. Is Not Available, Assign And Engrave G.T.I. I.D. No. (please refer to instrument and equipment log book for assignment of proper and sequential I.D. No.)

Condition on Receipt (visual): ______________________________

____

____

Missing Items: ______________________________________

____

____

Description of Damage (if any): ______________________________

____

____

Attach copy of packing slip if available.

Figure 8-1

Q/C Form No. 80-2
Page 2 of 2

ITEM	SERIAL NO.	ITEM	SERIAL NO.

ITEM	ASSIGNED GTI I.D. NO.	ITEM	ASSIGNED GTI I.D. NO.

Figure 8-2

collected samples or data for verification of validity. In most cases, items are identified using manufacturer serial numbers. In the absence of this labelling information, items are engraved with unique identity numbers assigned by GTI personnel. This activity is usually performed at the time of inspection but may be delayed until the initial specifications check (described in Section 9.1.8) is completed by qualified members of the technical staff responsible for the use of the item.

In the absence of manufacturer serial numbers, GTI I.D. numbers are assigned in accordance with the following discipline prefix codes:

Discipline	Discipline Prefix Code
air	A
biology	B
meteorology	M
soil, sediment	S
water	W
general	G

The I.D. numbers will begin with the prefix code and be followed by sequential numerical assignments in ascending order (i.e., for air, I.D. numbers will begin with A-1 and continue sequentially to A-100 or more, if necessary).

A logbook containing the equipment descriptions and assigned GTI I.D. numbers is maintained by the GTI QA Coordinators onsite. To prevent replication of I.D. numbers, shipment inspectors must obtain allowable codes from a GTI QA Coordinator prior to the assignment.

8.2 HISTORY RECORDS

8.2.1 Documentation and Record Forms

Maintenance of completed records for instrumentation and equipment is an integral part of GTI's quality control program. Documentation of historical information is accomplished through the use of an Instrument and Equipment History Record (QC Form 80-3) shown in Figures 8-3 and 8-4. This form provides a centralized location for recording the following vital information:

- Instrumentation and equipment identity and source information
- Calibration, operational check and maintenance protocols to be followed for each item
- Frequencies required for performance of QC check procedures
- Locations (forms, logbooks) for recording calibration and other information for each item.
- Summaries of initial instrument performance checkout results
- Descriptions of operational histories including provisions for recording narratives on malfunction, failure, repair/corrective action, and results.

8.2.2 Malfunction, Non-conformity, and Failure Events

Results of routine calibrations, maintenance or field/lab operational checks, normally recorded on data sheets or in logbooks, are not entered on the Instrument and Equipment History Form. In the event of a unit failure, malfunction, or indication of non-conformity (as a result of routine calibration), the unit is removed from service and marked with a tag indicating

GEOMET TECHNOLOGIES, INC.

Instrument and Equipment History Record

QC form 80-3
Page 1 of 2

ITEM: ____________________

MANUFACTURER: ____________________ GTI I.D. No: __________

VENDOR /SUPPLIER: __________ MODEL No. __________ MFR. S/N: __________

MFR: SPECIFICATIONS: __________ P.O. No. __________ DATE RECEIVED: __________

CALIBRATION, MAINTENANCE, AND CHECK REQUIREMENTS

MEASUREMENT PARAMETER	GTI I & E QC PROC. NO.	DESCRIPTION OF PROCEDURE AND STANDARDS	PROCEDURE REFERENCE	FREQUENCY REQUIRED	LOCATION/FORM FOR DATA RECORDING

Figure 8-3

QC form 80-3
Page 2 of 2

GEOMET TECHNOLOGIES, INC.

Instrument and Equipment History Record

ITEM:______________________ GTI I.D. No.: ______________ MFR. S/N: ____________

DATE/TIME	DESCRIPTION OF CALIBRATION/MALFUNCTION/PROBLEM/SERVICE/REPAIR	FUNCTIONAL STATUS	ACTION REQUIRED	NOTED OR PERFORMED BY (signature)

Figure 8-4

"INOPERATIVE-DO NOT USE". A narrative entry describing the problem is made on the Record. Corrective action is implemented and a subsequent notation is entered. Upon completion of the corrective action, the performance check is again performed and the results recorded as an additional entry. Successful completion of the performance check will result in a return of the item to use.

Procedures for troubleshooting are incorporated into the individual calibration, operational check, and maintenance methods.

8.3 QUALITY CONTOL/DATA FILING SYSTEM

Due to the relatively large number of quality control operations associated with the use of instruments and equipment on this study, considerable documentation will be generated. Efficient management of those records is necessary for retrieval and verification purposes. Two types of files will be used for systematic storage of the data generated to substantiate quality control of instruments and equipment. They are described in the following sections. The files will be maintained at the GTI field office near the Love Canal site.

8.3.1 General Files

A set of general files is used for the storage of data and documents pertaining to overall usage of instruments and equipment. Items included in this repository are shown in Table 8-1.

8.3.2 Individual Instrument and Equipment Item Files

This filing system is composed of individual files, one distinct file for each instrument and equipment item. Files for multiple items of the same type (i.e. high volume samples or Dupont P-125 air samplers) are grouped together in order of ascending serial or GTI I.D. number. General contents for each file are specified in Table 8-2. It is not necessary for all items in the table to be placed in each individual file. For example, instruction manuals for P-125 samplers need not be physically placed in each sampler file (since over 100 of these items are used) as long as a sufficient number of manuals are stored at the beginning of the grouping of the sampler files. Documents absolutely necessary for storage in every individual item file are marked with an asterisk (*) in the table.

Table 8-1

DOCUMENT STORAGE REQUIREMENTS FOR GENERAL FILES

Item No.	Description	Quantity
1-1	GCA-Quality Assurance Project Plan for multimedia monitoring at Love Canal-Appendix A Sampling Procedures	5 copies
1-2	GTI-Quality Control and Assurance Manual for multimedia monitoring at Love Canal	5 copies
1-3	Files containing all Shipment Receipt Records (QC Form 80-2) and packing slips - arranged in chronological order of receipt	As required
1-4	Instrument and Equipment Logbook. Logbook showing all equipment used on the study with serial numbers, assigned GTI I.D. numbers and date of receipt	1
1-5	Training Documentation forms (QA Form 80-1)-only when training involves use of specific instuments or equipment items	1 for each training session

Table 8-2

DOCUMENT STORAGE REQUIREMENTS FOR INDIVIDUAL INSTRUMENT AND EQUIPMENT ITEM FILES

Item No.	Description	Quantity
2-1*	Instrument and Equipment History Record (QC Form 80-3) to be completed for every individual instrument and equipment item used on the study	1 or more per item, depending on recording space needed
2-2	Manufacturers Instruction/Operation Manual	2 copies (minimum)
2-3	Instrument and Equipment Quality Control Procedure (QC Form 80-4) to be completed for all calibration, maintenance, and operational check procedures (1 procedure per form)	1 form for each procedure for each item
2-4*	Photocopies of all instrument/equipment calibration sheets completed during the study. If logbooks are used for recording calib. data, add photocopies of pages where calibration data appear	1 copy each as required
2-5	Original copies of NBS or manufacturers certificates of calibration received on delivery or at anytime during the project	1 or more as appropriate
2-6	Corrective Action Request Forms (QC Form 80-1) only when the request for action involves a specific instrument or equipment item. (Supply photocopy)	As appropriate

---

* Items necessary for placement in every individual file.

9.1 QUALITY CONTROL PROCEDURES FOR INSTRUMENTATION AND EQUIPMENT

9.1.2 Introduction

The systematic protocols employed by GTI to regulate instrument and equipment performance are described in this section. Control of performance is accomplished through the application of calibration, operational check and maintenance procedures to measurement equipment at specific intervals. Accepted physical standards are provided for accurate calibration while sufficient replication of tests is specified to ensure reliability. A description of the procedures appears in this section and the detailed QC procedures are included at the end of this section.

9.1.3 Quality Control Procedures

Procedures for the control of instrument and equipment performance are divided into three (3) categories as presented below:

- Calibration procedures
- Operational checks
- Maintenance protocols

Details of each type of procedure are described in the following sections. Troubleshooting and corrective action procedures are an important part of the quality control program for instruments and equipment. For the purpose of expediency and logistics, corrective action procedures have been incorporated into the individual QC procedures for calibration, operational checks, and maintenance.

All QC procedures are documented on the Instrument and Equipment Quality Control Procedure form (QC Form 80-4) which appears in Figure 9-1. Due to the relatively large number of procedures used in this study, a coding and identification system is used to uniquely identify the procedure and the sampling discipline with which it is associated. This coding and identity system, detailed in Table 9-1, also permits referencing on the Instrument and Equipment History form. In order to ensure that unique codes are assigned to each procedure, records of procedure number assigned are tracked on Instrument and Equipment QC Procedures Log Forms (QC Forms 80-7, 80-8, and 80-9) shown in Figures 9-2, 9-3, and 9-4. These forms also contain provisions for recording revisions to the procedures.

9.1.4 Calibration QC Procedures

Calibration is the process of establishing the relationship between instrument and equipment response and that of an accepted standard. Calibration procedures may be categorized as passive or dynamic depending on the operational characteristics of the measurement device. Passive calibrations involve the comparison of a measurement device output or reading to that of a standard with no provision for mechanical or electronic adjustment of the output of the instrument. Standard practice for passive calibrations is to

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No:____________________ Effective Date____________________

Procedure Type: Calibration______ Operational Check______ Maintenance______

Instr./Equip. Item:____

Manufacturer:__________________________________ Model No:_______________

Procedure Reference:____

PROCEDURE

Figure 9-1

Table 9-1

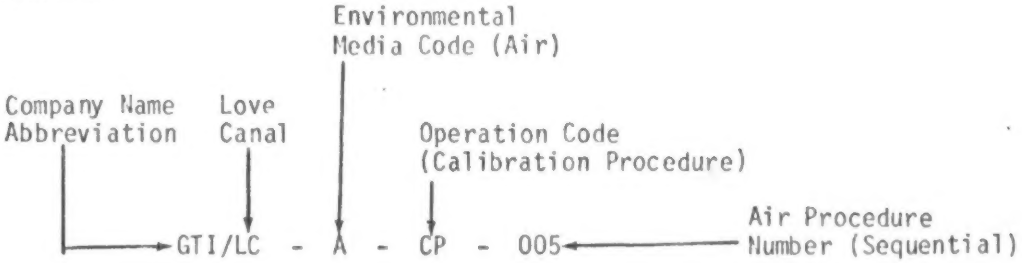
Identify Codes for Instrument and Equipment Quality Control Procedures

Environmental Equipment and Media	Code Letter
air	A
biological	B
meteorology	M
soil, sediment	S
water	W

EXAMPLE:

QC Procedure	Code Letter
calibration	CP
operational check	OP
maintenance	MP

EXAMPLE:


Environmental
Media Code (Air)
Company Name
Abbreviation
Love
Canal
Operation Code
(Calibration Procedure)
Air Procedure
Number (Sequential)
GTI/LC - A - CP - 005

GEOMET Technologies, Inc.

INSTRUMENT AND EQUIPMENT QC PROCEDURES LOG

Calibration

Q.C. FORM NO. 80-7

Proc. No.	Description	Date	Revision

Figure 9-2

GEOMET Technologies, Inc.

INSTRUMENT AND EQUIPMENT QC PROCEDURES LOG

Operational Checks

Q.C. FORM NO. 80-8

Proc. No.	Description	Date	Revision

Figure 9-3

GEOMET Technologies, Inc.

INSTRUMENT AND EQUIPMENT QC PROCEDURES LOG

Maintenance

Q.C. FORM NO. 80-9

Proc. No.	Description	Date	Revision

Figure 9-4

determine a correction factor for the device in cases where the output deviates from the reference standard. Dynamic calibrations imply that adjustments in the measurement device can be performed to render the instrument output identical to the response of the standard at the point of calibration. In this study, GTI anticipates using both passive and dynamic calibration procedures. Additionally, calibration check procedures will be implemented for some field instruments. These checks involve a comparison between instrument output and 1) an accepted standard or 2) another instrument calibrated against an accepted standard.

Measurement range linearity is an important consideration in the formulation of calibration procedures for instruments and equipment. GTI intends to address this requirement by specification of multipoint calibration values for test devices to cover the expected range of values to be measured.

Reliability of data measured using instrument or equipment devices depends to a large extent on the accuracy and reliability of the reference standards used in the calibration process. It is often possible to apply a range of standards differing in accuracy to the calibration of measurement devices. It is the intention of GTI to establish National Bureau of Standards (NBS) traceability wherever possible for reference standards employed during the course of the study. It must be recognized that NBS-traceable standards do not exist for every conceivable calibration standard or test.

For situations where NBS-traceability is not possible or is extremely difficult, GTI plans to use reference standards or materials accepted by regulatory agencies, research groups, or credible scientific organizations.

Due to the extensive facilities and equipment required for certain calibrations, GTI will not be able to perform some of the calibrations required. Under these circumstances, GTI will have the calibration performed by the manufacturer or at a reputable organization with proper facilities. In either case, a certificate of calibration for the devices will be obtained and kept on file for review.

The nature and stability of the monitoring equipment coupled with the required measurement accuracy will determine the frequency interval for calibration performance. For this aspect of calibration GTI will follow the practices recommended by government regulatory agencies, reputable scientific organizations, and instrument manufacturers. In the absence of recommended intervals, the technical staff at GTI will determine the necessary frequency from collective experience in the use of monitoring instruments.

Development of Calibration QC Procedures will be the responsibility of each GTI Task Manager. Detailed stepwise methods will be recorded on the Instrument and Equipment QC Procedures Form (QC Form 80-4). For each method, the Task Manager will be required to provide the following information:

- Reference standards to be used
- Environmental or other conditions necessary
- Data to be recorded

- Formulas for calculation of results
- Acceptability limits - criteria for assigning satisfactory or unsatisfactory performance
- Troubleshooting procedures - action to be taken if calibration results are beyond acceptability limits.

An abbreviated summary of the calibration procedures to be used during the study is presented in matrix form in Tables 9-2 and 9-3 while the detailed procedures are included in at the end of this section.

9.1.5 Operational Check QC Procedures

In order to ensure proper instrument performance in the field, operational procedures will be executed by GTI sampling personnel. The check procedures are intended to 1) provide verification of performance control between calibration periods and 2) prevent losses of samples or acquired data due to measurment or collection equipment failure. Performance of the operational checks may be immediately prior to sampling, during sample or data acquisition, or following the termination of sampling activities.

Operational check procedures may vary considerably with respect to the calibration procedures described in Section 9.1.4. Many of the procedures developed consist of observations, comparisons, or other tasks to verify proper performance and consequently system reliability. In some cases, the operational check may actually be a field calibration check performed with less rigor than the formal calibration executed under laboratory conditions.

Table 9-2

AIR SAMPLING INSTRUMENT AND EQUIPMENT CALIBRATION QC PROCEDURES

Instrument Equipment Item	Calibration and Standard(s)	Frequency	Control Limits	Corrective Action Planned
1. Dupont P-125 Air Sampler	1A) Flow rate calibration system and bubble tubes (GTI-LC-A-CP-008)	Before and after each sampling period	1A-1) Flow rate range 26-30 cc/min.	Repair or correct at the field offic or remove pump fro service
			1A-2) Replicate bubble tube runs must be within ±5% of mean flow rate	"
	1B) Flow rate calibration as above using increased pressure drop simulating media loading (GTI/LC-A-CP-008)	"	1-B) Flow rate with increased resistance must be within ±5% of mean flow rate	"
2. Dupont P-4000 Air Sampler	2A) Flow rate calibration with Dupont calibration system and bubble tubes (GTI/LC-A-CP-009)	"	2A-1) Flow rate range 1270 ±5% cc/min.	"
			2A-2) Replicate bubble tube runs must be within ±5% of mean flow rate	

Table 9-2 (Cont.)

AIR SAMPLING INSTRUMENT AND EQUIPMENT CALIBRATION QC PROCEDURES

Instrument Equipment Item	Calibration Standard(s)	Frequency	Control Limits	Corrective Action Planned
2. Dupont P-4000 Air Sampler	2B) Flow rate calibration as above using increased pressure drop simulating media loading (GTI/LC-A-CP-009)	Before and after each sampling period	2-B) Flow rate with increased presistance must be within ±5% of mean flow rate	
3. High Volume Air Sampling System				
A) Sampler (Flow Recorder)	3A) Flow rate calibration with orifice system (GTI/LC-A-CP-007)	On receipt, after major maintenance and at study completion	3A) $Q_o - Q_c \leq 0.05\ Q_c$ Q_o=Observed flow rate Q_c=Calib. curve flow rate	Rerun points for which the control limit is exceeded until acceptance is obtained
B) Elapsed Time Meter	3B) Time accuracy check against Base reference chronmeter (GTI/LC-A-CP-004)	At start and completion of study	3B) ±2 min/24hrs.	Adjust or replace
C) On/Off Timer	3C) Tripping accuracy check against elapsed time meter	At start and completion of study	3C) ± 15min/24hrs.	Adjust or replace

Table 9-2 (Cont.)

AIR SAMPLING INSTRUMENT AND EQUIPMENT CALIBRATION QC PROCEDURES

Instrument Equipment Item	Calibration Standard(s)	Frequency	Control Limits	Corrective Action Planned
4. Base Reference Chronometer	4A) Check synchronization against U.S.N. Observatory Master Clock (GTI/LC-A-CP-002)	1x/day	4A) ± 1sec/ synchronization	repeat synchronization to attain acceptability
	4B) Check accuracy against U.S.N. Observatory Master Clock (GTI/LC-A-CP-002)	1x/day	4B) ± 1sec/24 hr. accuracy	Adjust or replace
5. Digital LCD Quartz Watches	5A) Synchronize against Base reference Chronometer (GTI/LC-A-CP-003)	At start of each sampling or 1x/day at end of each sampling	5A) ± 2sec. synchronization	Repeat to achieve synchronization
	5B) Check accuracy against Base reference Chronometer (GTI/LC-A-CP-003)	At end of each sampling	5B) ±10sec/12hrs.	Replace
6. Stopwatches	Check time accuracy against Base reference Chronometer (GTI/LC-A-CP-006)	At beginning and end of study or in case of accidental mishandling	±0.6sec/10min (0.1%)	Replace

Table 9-2 (Cont.)

AIR SAMPLING INSTRUMENT AND EQUIPMENT CALIBRATION QC PROCEDURES

Instrument Equipment Item	Calibration and Standard(s)	Frequency	Control Limits	Corrective Action Planned
7. Bubble Tubes and Burets	Check volume accuracy with water displacment (GTI/LC-A-CP-001)	At beginning of study	± 1% for 50ml tubes	Use empirical correction factor

Table 9-3

TRANSPORT STUDY AND METEOROLOGICAL MONITORING CALIBRATION QC CHECKS

	Instrument or Equipment Item	Calibration and Standard(s)	Frequency	Control Limits	Corrective Action Planned
1.	Reference Thermometer (ASTM 63F, 64F)	Multipoint calibration by manufacturer against NBS standard test	At time of purchase	±0.2°F	Replace if breakage occurs
2.	Hygrothermograph	A) Temperature checked at 2 points against reference thermometers (GTI/LC-M-CP-005)	At start and completion of study	±1°C (±2°F)	Repeat calibration or replace unit if necessary
		B) RH checked at 2 points against sling or asperated psychrometer (GTI/LC-M-CP-004)	At start and completion of study	±3%RH(20-80%RH)	Recalibrate in lab facilities or replace if necessary
3.	Aspirated Psychrometer	Temperature checked at 2 points against reference therometers (GTI/LC-M-CP-006)	Monthly	±0.5°C	Replace thermometer if accuracy exceeds control limits
4.	Wind Speed/Direction Measurement System	A) Wind Speed-calibrated by manufacturer against a primary standard (GTI/LC-M-CP-002)	At start and completion of study	±2%	Make appropriate compensation during data reduction

Table 9-3 (Cont.)

TRANSPORT STUDY AND METEOROLOGICAL MONITORING CALIBRATION QC CHECKS

Instrument or Equipment Item	Calibration and Standard(s)	Frequency	Control Limits	Corrective Action Planned
4. Wind Speed/Direction Measurement System	B) Wind Direction-Align true north against well-documented true north landmarks. Check 3 other directions on chart (GTI/LC-M-CP-002)	At start and completion of study	±3° azimith	Adjust transducer assembly or replace unit
5. Rain Gauge	Check digital accumulator with water equivalent to 1 inch precipitation (GTI/LC-M-CP-001)	At start and completion of study	±0.02 inches accuray	Make repairs or adjustments in the field-replace if not corrected.

GTI Task Managers will be responsible for development of the operational check procedures. The specific details of each procedure are recorded on the Instrument and Equipment Quality Control Procedure sheets (QC Form 80-4) and will include the following information:

- Reference standards for comparison
- Environmental or other conditions necessary
- Data to be observed or recorded or task to be performed
- Formulas for calculation of results
- Acceptability limits - criteria for assigning satisfactory or unsatisfactory performance
- Troubleshooting procedures - action to be taken if operational check results are beyond acceptability limits.

A summary of the operational check procedures to be implemented in the field appears in Tables 9-4 through 9-6. Detailed check procedures are included at the end of this section.

9.1.7 Maintenance QC Procedures

Maintenance procedures are tasks executed at periodic intervals to ensure the mechanical and electronic integrity of instruments and equipment. In general, these procedures may involve one or more of the following operations:

- disassembly
- cleaning
- lubrication
- replacement of worn parts
- minor adjustments
- reservoir filling (ink, water, oil, etc.)

Table 9-4

AIR SAMPLING OPERATIONAL QC CHECKS

Parameter Pollutant Class	Operational Check	Frequency	Control Limits	Corrective Action Planned
1. Volatile Organics (Dupont P-125/Tenax)	a) Battery check	At site, prior to startup	a) Battery check LED should light	Use backup pump
	b) Low flow detector (no load)	"	b) Low flow detector LED should not light	"
	c) Low flow detector (load imposed)	"	c) Low flow detector LED should light	"
	d) Pump operation	At site, just after sampling start	d) Pump should operate	"
	e) Low flow detector	"	e) Low flow detector LED should not light	"
	f) Flow rate check (GTI/LC-A-OP-001)	At site, just prior to terminating sample	f) Low flow detector LED should not light	Make notation on sampling sheet, sample tag chain of custody form
2. Semivolatile Organics and Pesticides (Dupont P-4000/P.U.F.)	Same as described for Volatile Organics (GTI/LC-A-OP-002)			
3. Particulates/Metals (Hivol Samplers/ G.F. Filters)	a) Flow recorder zero	At site prior to startup	a) Chart should be set at zero	Adjust
	b) Initial Flow rate check	At site, 5 min. after startup	b) Initial stabilized flow should be within ±10% of established initial flow	Use backup system

Table 9-4 (Cont.)

AIR SAMPLING OPERATIONAL QC CHECKS

Parameter Pollutant Class	Operational Check	Frequency	Control Limits	Corrective Action Planned
3. Particulates/Metals (Hivol Samplers/ G.F. Filters)	c) Flow rate range	At site, upon retrieval	Mean flow rate should be between 30-60 C.F.M.	Make notations
	d) Filter conidition (GTI/LC-A-OP-002)	At site, upon retrieval	No evidendence of leakage	Make notations

Table 9-5

TRANSPORT STUDY AND METEOROLOGICAL MONITORING OPERATIONAL QC CHECKS

Parameter(s) or Pollutant(s) Measured	Operational Check	Frequency	Control Limits	Corrective Action Planned
1. Temperature-Outdoor (Hygrothermograph)	Check against aspirated psychrometer thermometer (GTI/LC-M-OP-004)	Weekly	±1.0°C (2°F)	1. Adjustment and recalibration 2. Determine exten of data affecte
2. Relative Humidity-Outdoor (Hygrothermograph)	Check against relative humidity determined with sling psychrometer (GTI/LC-M-OP-004)	Weekly	±3%R.H.	1. Recheck 2. If outside limi recalibrate in field lab
3. Relative Humidity-Indoor (Aspirated Psychrometer)	1. Damaged thermometers 2. Resevoir water level 3. Fan operation (GTI/LC-M-OP-005)	1. During each use 2. " 3. "	1. Visual inspectio 2. " 3. "	1. Replace 2. Refill resevoir 3. Replace batteri
4. Wind Speed (Anemometer)	Readout to be checked for reasonability against visual observations of existing meteorological conditions (GTI/LC-M-OP-002)	Daily	Reasonable correlation	Calibration check i observations warran action
5. Wind Direction (Wind sensor and recorder)	Readout to be checked for reasonability against visual observations of existing meteorological conditions (GTI/LC-M-OP-002)	Daily	Reasonable correlation	Calibration check i observations warran action

Table 9-5 (Cont.)

TRANSPORT STUDY AND METEOROLOGICAL MONITORING OPERATIONAL QC CHECKS

Parameters(s) or Pollutant(s) Measured	Operational Check	Frequency	Control Limts	Corrective Action Planned
6. Precipitation (Tipping-bucket rain gauge)	1. Readout to be checked against existing meteorological conditions	Daily	Reasonable correlation	Calibration check if observations warrant action
	2. Visual check for gross response (GTI/LC-M-OP-001)	During or after rainfall events (depending on time of occurance)	Proper response judged visually	Free blocked bucket or repair poor gauge contact. Determine extent of data affected

Table 9-6

BIOMONITORING OPERATIONAL QC CHECKS

Parameter(s) or Item(s)	Operational Check	Frequency	Control Limits	Corrective Action Planned
1. VICTOR-Mouse and Rat Traps	Test tripping mechanism	Before setting	Proper tripping of bait pedal	Repair or replace
2. HAVE-A-HEART Live Traps	"	"	"	"
3. Trap Line inspection	Survey of trap line for security	Hourly	Trap line must not be disturbed	Make notation on field data sheets, sample tag, chain of custody form

Due to the relatively short duration of this study, it is anticipated that there will be relatively few maintenance events. The equipment used will be primarily new and is generally designed to be maintenance-free for extended periods of time.

In the event that significant maintenance is required for a measurement device, the procedure will be documented by the Task Manager on the Instrument and Equipment QC Procedure sheet (QC Form 80-4) prior to performance. The maintenance procedure will generally be derived from the manufacturer's operating manual.

In order to prevent or minimize downtime due to failure, the GTI staff has identified components for instruments that are subject to wear and have provided an appropriate inventory of spare parts. In addition, approximately 10 percent of the required air sampling instruments are on reserve as backup units.

The GTI Task Managers will identify a list of manufacturers capable of effecting repairs, providing parts for instruments, or rendering assistance in the event of a malfunction.

9.1.8 Initial Performance and Specifications Check

Each item of instrumentation or equipment is subject to an initial performance/calibration check prior to first use. The purpose of this check is: (1) to verify that the instrument is performing properly and (2) that it meets the manufacturer specifications or those criteria necessary for the study. Raw data for the initial calibration or check is recorded in serialized calibration logbooks or on calibration data sheets. The procedures used for the performance check are generally those specified by government regulatory agencies, the manufacturer of the item or by the GTI staff. Results of the initial performance check are then recorded in narrative form on the item's Instrument and Equipment History Record. The entries will include the following information:

- Specifications required
- Results obtained (compared to specifications required)
- Indication of conformity or non-conformity

Photocopies of the raw data from logbooks or data sheets will be attached to the record to substantiate performance levels achieved.

Systems not conforming to required specifications require corrective action. In the case of non-conforming items, the following action is required:

- Correction/repair by GTI personnel
- Return to the manufacturer or vendor for repair or replacement
- Repetition of the initial performance check after repair or replacement.

10.1 PERFORMANCE OF INTERNAL AUDITS

Purpose: a systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) performance audits in which quantitative data are independently obtained for comparison with routinely obtained data, or (b) system audits of a qualitative nature which consist of an on-site review of the quality assurance sytem, and its physical facilities for sampling, calibration, and measurement.

10.1.1 Performance Audits

An independent performance audit refers to the procedures used by the Task Manager to evaluate the quality of data produced by the total sampling system. It provides a means of assessing data collected by several field teams and/or air surveillance networks with the potential of identifying biases or excessive variations in the data collection procedures. A performance audit will not only provide an independent quality check, but will also identify the weak points in the measurement system. Performance audits are normally a quantitative appraisal of quality. Thus the Task Manager, an individual chosen for this background knowledge of measurement system, will be able to guide field teams in using improved techniques. In addition, this individual's role will be one of aiding the quality function of all activities for which he is responsble, utilizing the cross-fertilization of good measurement techniques to improve the quality of the collected data.

Unique problems are expected when implementing an auditing program for air quality monitoring systems. One of the first is defining what consitutes an item in the auditing process. This approach obviously requires support from routine equipment checks and operational procedures to verify that no malfunctions occurred between audits. However, these audits are performed independent of and in addition to normal quality control checks by the operator. Independence will be achieved by having the audit made by a different operator who will use a separate set of calibrated flowmeters and reference standards to check the sample collection system:

a. Flow rate devices

b. Instrument calibration

After the auditing schedule has been implemented, the audits results will be plotted on control charts to give the Task Manager a visual picture of changes in the performance so that corrective actions may be taken when necessary.

10.1.2 System Audits

A system audit is an on-site inspection and review of the quality assurance system used during this project. Such an audit is scheduled and performed at any time during the life of the project but most probably will be conducted after monitoring has been initiated. This audit may be supplemented by additional audits for one or more of the following reasons:

- Significant changes are made in operating Procedures Manual
- It is necessary to verify that corrective action has been taken on a nonconformance report in a previous audit.
- Additional or special audits are required by the audit procedures for a specific report.
- A request is made by the Project Manager.

Some of the items that will be audited are listed below:

1. Organizational and responsibility - Is the quality assurance organization operational?
2. Sample Collection - Are written procedures for collection available and are these followed as written?
3. Calibration - Are written procedures available and are they followed? In addition, a review will be made of procedures used to establish traceability of calibration standards to standards of higher accuracy.
4. Audits - Are control charts for performance audits reviewed by management?
5. Chain-of-Custody - Have the appropriate steps been followed in the traceability of sample origin and integrity?
6. Operational Procedures - Are the appropriate Q.C. checks made in the field and are records maintained of these checks?
7. Corrective Action - Is the appropriate chain of command followed in responding to out-of-control situations? Are these situations properly reported on the Corrective Action Record Form?
8. Health and Safety - Are the proper precautions taken to protect sampling team during field operation?
9. Preventative Maintenance - Are the preventative maintenance procedures followed as recommended in Q.A. plan.

10. Are proper records and logs maintained of all control documents?

Prior to audit, the Q.A. Manager notifies the Project Manager and Task Managers of the unit to be audited, of the proposed time and place of the audit. Audits are performed on the basis of written checklists (Figure 10.1) or lists of questions prepared prior to the audits. During the conduct of the audit each item on the list is marked with one of the following entries: CI = closed item; OI = open item; and NA = item not applicable. Open items found during an audit will be listed in the audit report which will be delivered to the Project Manager and the Task Manager whose unit is being audited. Such audit reports will be prepared by the auditor no later than 3 days from the day of the audit and will require response from the Task Manager within a similar time frame. All open items will need to be addressed by detailed explanation of remedial action which needs to be taken. Open items which receive proper remedial action will be closed by the auditor, items which are left open will be in violation of the Quality Assurance Project Plan.

Sheet __ of __

AUDIT CHECKLIST

TITLE: Multimedia Monitoring At Love Canal (LC-1-619 026)
Attachment to Audit No. LC _______.

Audit Approved by: ____________________ Date ____________________
Q.A. Manager

Audit Approved by: ____________________ Date ____________________

Documents used as reference during audit: ____________________

____

Document Reference	Activity Audited	Findings

Figure 10-1

11.1 REPORTS TO MANAGEMENT

Purpose: to keep management informed by submitting updated reports in order that they may make the appropriate managerial decisions.

11.1.1 Principles of Reporting to Management

The following are general principles considered:

The Quality Assurance Officer will discuss with Task Managers, Field Coordinators, and the Program Manager what reports need to be published on a regular basis to provide information required for current decision-making. Reports will be reviewed by the GTI Managerial Review Committee which will meet every two weeks with J. Moran, GTI Program Director, and Dr. Mason, Project Manager. This meeting will occur typically at GTI Corporate headquarters in Gaithersburg, Maryland, but alternatively at Niagara Falls, should it be necessary to review technical performance and problems. Other members of the Managerial Review Committee are Messrs. Swift, Nagda, Malloy, and Moschandreas, senior members of GTI's managerial staff.

The reports will include:

- The percentage of replicate samples taken during the study.
- Instrument and equipment downtime.
- On-site OSH inspection results.
- Assessment of measurement data accuracy and precision.

- Significant QA problems and recommended solutions.
- Results of system audits performed during the study by QA officer.
- Corrective Action Reports.
- Percentage of samples collected versus total samples expected.

Reports will be presented in summary format. When not presented graphically, the report should provide abstracts of essential points. These reports will compare current data with some base when possible. The basic standard of comparison will probably be based on previous performance (history). When applicable, quality control charts will be used to report results from performance audits.

Q.C. Form No. 80-7
Air

GEOMET Technologies. Inc.

INSTRUMENT AND EQUIPMENT QC PROCEDURES LOG

Calibration

Proc. No.	Description	Date	Revision
GTI/LC-A-CP-001	Bubble Tubes and Burets: Volume Calibration	8/14/80	0
GTI/LC-A-CP-002	Base Reference Chronometer: Calibration	8/26/80	0
GTI/LC-A-CP-003	Digital LCD Quartz Watches: Calibration	8/26/80	0
GTI/LC-A-CP-004	Elapsed Time Meters (High Vols): Calibration	8/26/80	0
GTI/LC-A-CP-005	On/Off Timers (High Vols): Calibration	8/26/80	0
GTI/LC-A-CP-006	Stopwatches: Calibration	8/26/80	0
GTI/LC-A-CP-007	High Volume Air Samplers: Flow Rate Calibration	8/26/80	0
GTI/LC-A-CP-008	DuPont P-125 Air Sampler: Flow Rate Calibration	8/28/80	0
GTI/LC-A-CP-009	DuPont P-4000 Air Sampler: Flow Rate Calibration	8/28/80	0

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-001 Effective Date 8/14/80

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: Soap Bubble Tubes and Burets

Manufacturer: Miscellaneous Model No:

Procedure Reference: U.S. EPA (1977) Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. II, Section 2.1.2, pp. 6-11

PROCEDURE

VOLUME CALIBRATION (GRAVIMETRIC VOLUME DISPLACEMENT METHOD)

(Record Data on Soap Bubble Meter Calibration Log [QC Form 80-5])

Step: DESCRIPTION

1. Secure stoppered containers of appropriate size to contain the volume of water to be used to calibrate the bubble tube or buret.
2. Determine the initial weight of each container to the nearest 0.001 grams and record.
3. Examine bubble tube for cleanliness. If necessary, clean with a soap solution and rinse thoroughly with deionized water.
4. Clamp the bubble tube upright with a stopcock connected to the end with the restricted aperture. Fill the tube with water and remove all air bubbles--rinse several times if necessary.
5. Fill the bubble tube with water such that the bottom of the meniscus is at the top volume marking.
6. Measure the temperature of the water in the tube with a mercury thermometer and record.
7. Drain the displacement volume into a tared container, immediately cap, and weigh to the nearest 0.001 grams.
8. Repeat Steps 5 through 7 two additional times and record all data.
9. Calibration of bubble tube volumes in excess of 100ml must be performed by weighing 100ml (or smaller) aliquots and calculating the desired displacement volume by addition of the sum of the aliquots. In the event that multiple aliquots are used to calibrate a large volume, the determination of each graduated interval must be performed in triplicate.
10. Obtain the density of water at the measured temperature from reference table on the calibration data sheet.
11. Calculate the mean displacement volume using the equations provided on the data sheet.

ACCEPTABILITY: Manufacturer stated displacement volume must not exceed ±1% of the volume determined by calibration.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-002 Effective Date 8/26/80

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: Base Reference Chronometer

Manufacturer: Seth-Thomas, Inc. Model No: 2574-0000

Procedure Reference: Seth-Thomas Instructions/GTI staff

PROCEDURE

CHRONOMETER CALIBRATION

Step: Description

1. The reference standard for calibration is the U.S Naval Observatory MASTER CLOCK. Reference signals for synchronization and calibration purposes are obtained through telephone contact.
2. To obtain the synchronizing time signal, the telephone number (202)-254-4950 is dialed. The time signals consist of four(4) verbal announcements of the time (to the nearest second) at 15-second intervals. These announcements preceed an auditory sound signal which marks the exact time announced. This system permits time synchronization to the nearest second.
3. To set the clock, pull the set knob on the back of the movement and rotate in either direction to advance or retard the clock mechanism.
4. Synchronize the chronometer as per Step 3 using the reference signals for time described in Step 2. Record all results.

ACCEPTABILITY: ± 1 second synchronization

5. Verify the accuracy of the timepiece by checking the time (as described in Step 2) at an interval of 12 or 24 hours. Record all results.

ACCEPTABILITY: ± 1 second in 24 hours (for accuracy)

6. In the event that periodic calibration checks show a deviation in excess of that stated in Step 5, repeat the synchronization. Record all results.
7. If subsequent periodic accuracy checks reveal deviations in excess of that stated in Step 5, the chronometer must be adjusted or replaced.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-003 Effective Date 8/26/80

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: Digital LCD Quartz Watches

Manufacturer: Armitron Company Model No: Quartz LC

Procedure Reference: Armitron Manual/GTI staff

PROCEDURE

CALIBRATION PROCEDURE FOR DIGITAL WATCHES

Step: Description

1. Use the BASE REFERENCE CHRONOMETER (calibrated against the U.S. Naval Observatory Master Clock) as the reference for all time calibrations. Synchronization is performed prior to each field sampling effort. Accuracy is checked at the conclusion of sampling.
2. To synchronize hours, press S2 three(3) times and S1 repeatedly to set.
3. To synchronize minutes, press S2 two(2) times and S1 repeatedly to set.
4. To synchronize seconds, press S2 one(1) time and S1 repeatedly to set.
5. Synchronize the watch according to Steps 1 through 4. Record all results.

ACCEPTABILITY: Synchronization to within ± 2 seconds of time indicated on base reference chronometer

6. Accuracy of the digital watches is checked upon completion of sampling. Check the time on the BASE REFERENCE CHRONOMETER and record the time indicated on the digital watch.

ACCEPTABILITY: ± 10 seconds in 12 hours (accuracy)

7. If the accuracy in Step 6 cannot be achieved, the watch is removed from service and replaced.

Appendix Q
Plan 3.1
QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-004 Effective Date 8/26/80

Procedure Type: Calibration X Operational Check ___ Maintenance ___

Instr./Equip. Item: High Volume Air Samplers - Elapsed Timers

Manufacturer: General Metals, RAC, Sierra & Others Model No: ___

Procedure Reference: U.S. EPA (1977) Quality Control Procedures for Air Pollution Measurement Systems

PROCEDURE

CALIBRATION OF ELAPSED TIME INDICATORS ON HIGH VOLUME AIR SAMPLERS

Step: Description

1. Set up timer for a manual ON/OFF sequence.
2. Record initial elapsed time indicator reading.
3. Using the Base Reference Chronometer (set against U.S. Naval Observatory Master Clock) start the elapsed time meter and record the starting reference time.
4. After an exact 24-hour operating period, shut off the elapsed time meter and record the ending reference time indicated on the Base Reference Chronometer.
5. Record the final elapsed time indicator reading.

ACCEPTABILITY: ± 2 minutes in 24 hours

6. If the indicator fails to give results within acceptable limits, adjust or replace the indicator and repeat Steps 1 through 5.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-005 Effective Date 8/26/80

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: High-Volume Air Samplers-ON/OFF Timer

Manufacturer: General Metals, RAC, Sierra, Etc. Model No: ____

Procedure Reference: U.S. EPA (1977) Quality Control Procedures for Air Pollution Measurement Systems

PROCEDURE

CALIBRATION CHECK OF ON/OFF TIMERS ON HIGH-VOLUME AIR SAMPLERS

Step: Description

1. Plug a correctly wired timer into an electrical outlet.
2. Set the timer to the correct time.
3. Set the ON and OFF TRIPPERS for a 24-hour test.
4. Plug the sampler into one of the outlet plugs and an elapsed time meter into the other.
5. Check the system by manually operating the switch on and off.
6. Start the on/off timer and cycle for a 24-hour sample duration. The elapsed time meters are used as the reference for time. The meter should be set to zero when the on/off timer operation is initiated.
7. When the cycle is completed, record the elapsed time from the elapsed time meter.

ACCEPTABILITY: ±15 minutes in 24 hours (compared to time from the elapsed time meter

8. If acceptability limits cannot be achieved, adjust the tripper switches and repeat Steps 1 through 7 or replace the timer.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-006 Effective Date 8/26/80

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: Stopwatches

Manufacturer: Miscellaneous Manufacturers Model No: ____

Procedure Reference: GTI Staff

PROCEDURE

STOPWATCH CALIBRATION

Step: Description

1. The BASE REFERENCE CHRONOMETER (calibrated against the U.S. Naval Observatory MASTER CLOCK) is the timepiece used as the reference standard for time measurements.
2. Set the stopwatch to read zero time.
3. Using the BASE REFERENCE CHRONOMETER time as reference, start the stopwatch and record the start time (to the nearest second) indicated on the BASE REFERENCE CHRONOMETER.
4. Contine timing for a minimum time of 10 minutes (600 seconds).
5. At the end of the interval, stop the stopwatch and record the ending time (to the nearest second) indicated on the BASE REFERENCE CHRONOMETER. Record the elapsed time measured by the stopwatch.

ACCEPTABILITY: ± 0.6 seconds in 10 minutes (0.1%)

6. Repeat Steps 2 through 5 two(2) additional times for verification.
7. If the results exceed the acceptable limits found in Step 5, the stopwatch must be removed from service and replaced.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-007 Effective Date 8/26/80

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: High Volume Air Samplers

Manufacturer: General Metals, RAC, Sierra or others Model No: ____

Procedure Reference: U.S. EPA (1977) Quality Control Procedures for Air Pollution Measurement Systems

PROCEDURE

CALIBRATION OF HIGH VOLUME AIR SAMPLER FOR FLOW RATE

Step:	Description
1.	Remove filter retaining plate from the sampler to be calibrated and deactivate the electronic flow controller by placing a sleeve over the sensing element.
2.	Attach the variable resistance orifice (VRO, BGI Inc., S/N#3703) to the sampler and position the orifice setting to full open. Secure the VRO fall plate to insure an air tight seal with the orifice gasket. Attach the slack tube manometer (General Metal Works #1211) to the sampler unit.
3.	Manually turn the sampler on and introduce a load on the system by closing the VRO halfway. Allow the sampler to operate for 5 minutes.
4.	Turn motor off and place a fresh chart on the unit. The chart should include the following information: high volume sampler identification, date and time of calibration, and operators name. The chart should be labelled "Calibration Data".
5.	Check the recorder for proper operation, and zero the pen if necessary.
6.	Determine 5 approximately equally spaced intermediate points which provide pressure drops between a maximum and minimum point and record the following data on the calibration sheet: a. pressure difference from the manometer (in. H_2O) b. flow rate indicated on Dickson recorder. Repeat three points centralized in the vicinity of the samples normal flow rate to insure accuracy in the field: (The Dickson Recorder should be tapped gently prior to reading, to insure that the recorder pen is in its final position.)

CALIBRATION OF HIGH VOLUME AIR SAMPLER FOR FLOW RATE (Cont.)

7. Record the air flow rate from the VRO hi-vol calibration curve for each flow recorder reading.

$$\text{ACCEPTABILITY} = 100\,\frac{(Q_o - Q_c)}{Q_c} \text{ within } \pm 5\%$$

Q_o = observed flow rate
Q_c = flow rate from calibrated curve

8. If the air flow rate exceeds the acceptable limits, rerun points for which percent deviation exceeds ±5% until acceptance limits are attained.

9. Correct the sample flow rate to standard conditions using the following formula:

$$Q_2 = Q_1\sqrt{\left(\frac{T_2\ P_1}{T_1\ P_2}\right)}$$

Q_2 = corrected flow rate (SCFM)
Q_1 = flow rate from chart (CFM)
T_2 = absolute temperature (298°K)
P_1 = barometric pressure when hi-vol was calibrated
T_1 = absolute temperature when hi-vol was calibrated
P_2 = standard barometric pressure (29.92 in. Hg)

(Prior to each calibration barometric pressure is recorded from radio, NOAA frequency 162.55 MH_z).

10. Plot a flow curve for the calibration by plotting Dickson chart indicator readings and the calculated SCFM points.
11. Label the calibration graph with date, time, high volume sampler I.D. number, and operators signature.

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-008 Effective Date 082880

Procedure Type: Calibration X Operational Check ______ Maintenance ______

Instr./Equip. Item: DuPont Constant Flow Sampler

Manufacturer: E.I. Dupont Model No: P-125

Procedure Reference: P-125 Operating Manual, GTI Staff

PROCEDURE

FLOW RATE CALIBRATION

Step: Description

1. Select a set of samplers for calibration and assemble necessary equipment.
2. Record the operator name, date, and time at the beginning of the calibration. Also record serial or GTI I.D. numbers of calibration kits, stopwatches, thermometers, bubble tubes, and pumps.
3. Measure the ambient air temperature with a mercury thermometer and obtain the water vapor pressure at the ambient temperature from appropriate tables. Obtain the barometric pressure from the NOAA radio station (frequency 162.55 MHz). Record all data.
4. Start the pump and check the battery condition by depressing the test button and observing the battery check LED.

ACCEPTABILITY: Battery Check LED should light

5. If the battery condition is unacceptable, place the pumps back on the charging rack and select another pump and repeat Step 4.
6. Place finger tightly over the pump inlet, wait 20-30 seconds and check the condition of the low flow indicator LED by depressing the test button.

ACCEPTABILITY: Low Flow Indicator LED should light

7. If results of Step 6 are unacceptable, mark the sampler with a tag indicating "INOPERATIVE-DO NOT USE", make an entry in the sampler's Instrument and Equipment History File (Q.C. Form 80-3). Remove the pump from service.
8. Record acceptable results from Steps 4 and 6 on the calibration sheet.
9. Allow the pump flow rate to stabilize and attach to the calibration system shown on the following page.

FLOW RATE CALIBRATION (CONT.)

10. Impose a 4±1 inches of water pressure drop on the Pressure Drop Meter by adjustment of the drop value. Allow sufficient time for the pump to stabilize. Record the exact pressure drop imposed.
11. Adjust the Flow Rate Meter to read midscale using the Flow Rate Valve.
12. Determine the initial flow rate of the pump using the bubble tube and record the time required for bubble travel between two appropriate volume markings on the tube. Calculate the flow rate (corrected to STP) as follows:

$$\begin{array}{c}\text{STP Flow Rate}\\ \text{(cc/min)}\end{array} = \left[\frac{\begin{array}{c}\text{Bubble Tube}\\ \text{Vol. Used (cc)}\end{array}}{\begin{array}{c}\text{Travel Time}\\ \text{(min)}\end{array}}\right]\left[\frac{Pf - Pv}{Ps}\right]\left[\frac{Ts}{Tf}\right]$$

where Pf = atmospheric pressure (in. Hg) from NOAA 162.55 MHz (Step 3)
Pv = water vapor pressure at the calibration temperature (Step 3)
Ps = standard atmospheric pressure (29.92 in. Hg)
Ts = standard temperature (293°K)
Tf = ambient temperature of calibration (°K) (Step 3)

13. Adjust the Flow Adjustment Screw on the sampler to achieve a flow rate between 26 and 30 cc/min. as determined by the bubble tube method. Once the target flow at STP has been achieved, verify three (3) times with the bubble tube and record all results. Record the position of Flow Rate Meter for these measurements. Calculate all STP flow rates according to Step 12. Calculate the mean flow rate by summing the STP flow rates from each of the three (3) runs and dividing by three (3).

ACCEPTABILITY: The Mean Flow Rate must be within the range of 26-30 cc/minute. The deviation of the individual flow rates from the Mean Flow Rate must not exceed ±5%

14. Increase the Pressure Drop Meter to read 23±1 in. H_2 and allow sufficient time for the pump to stabilize.
15. Determine the flow rate at the increased resistance with the bubble tube.

ACCEPTABILITY: ±5% Maximum Difference in flow rate from the mean flow rate determined in Step 13

16. If results in Step 15 are unacceptable, reject the pumps and remove from service.
17. If results are within acceptable limits, open the Pressure Drop Valve until the Pressure Drop Meter reads 3±1 in. H_2O. Turn pump off.
18. Mark all acceptable pumps with blue sticker and return to charging rack.

QC Form 80-4

Appendix Q
Plan 3.1

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-008 Effective Date 082880

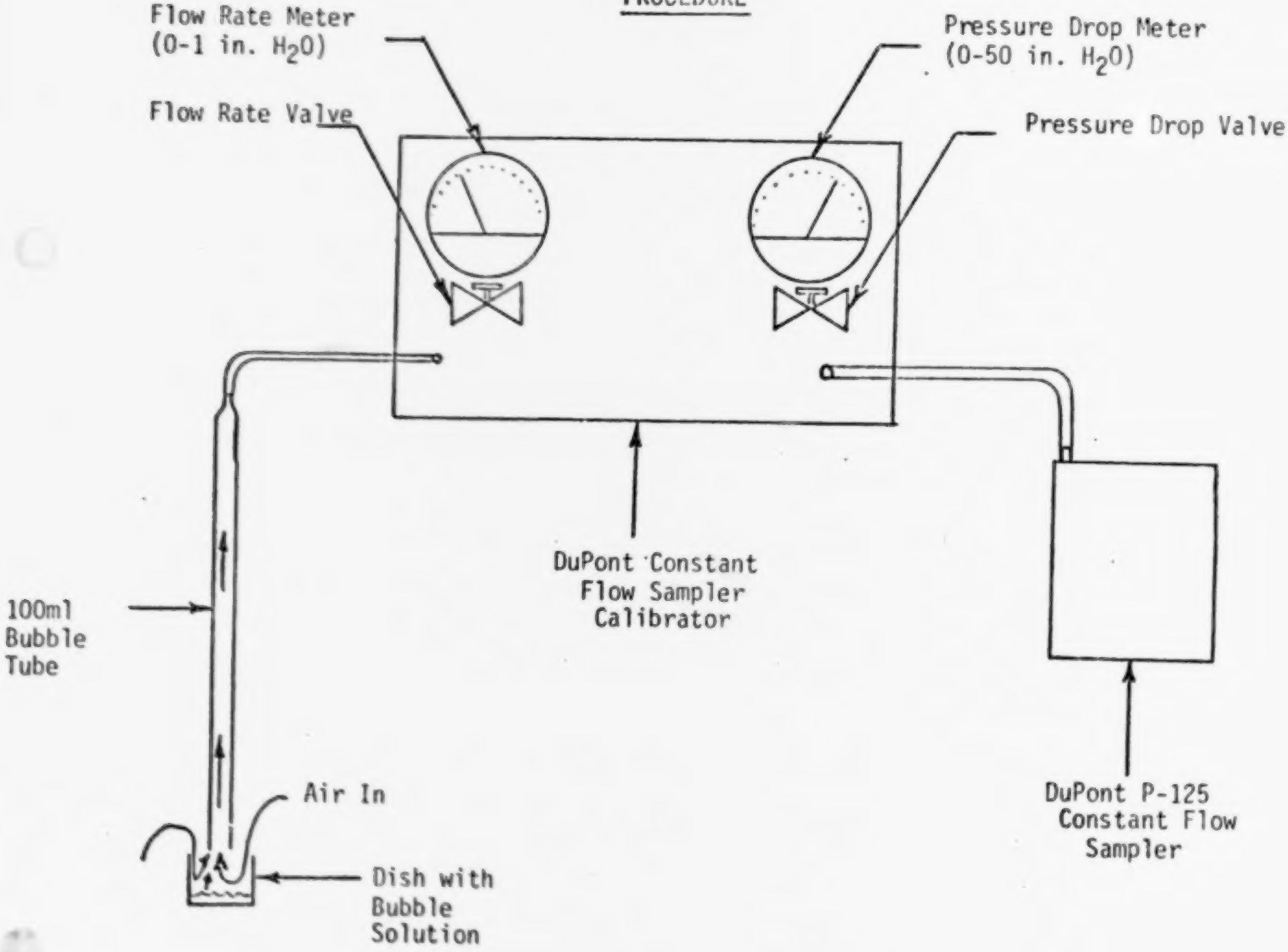
Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: Dupont P-125 Constant Flow Sampler

Manufacturer: E.I. Dupont Model No: P-125

Procedure Reference: P-125 Operating Manual/GTI Staff

PROCEDURE


Flow Rate Meter
(0-1 in. H_2O)
Pressure Drop Meter
(0-50 in. H_2O)
Flow Rate Valve
Pressure Drop Valve
DuPont Constant
Flow Sampler
Calibrator
100ml
Bubble
Tube
Air In
Dish with
Bubble
Solution
DuPont P-125
Constant Flow
Sampler

CALIBRATION CONFIGURATION FOR DUPONT P-125 SAMPLERS

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-009 Effective Date 082880

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: DuPont Constant Flow Sampler

Manufacturer: E.I. Dupont Model No: P-4000

Procedure Reference: P-4000 Operating Manual, GTI Staff

PROCEDURE

FLOW RATE CALIBRATION

Step: Description

1. Select a set of samplers for calibration and assemble necessary equipment.
2. Record the operator name, date, and time at the beginning of the calibration. Also record serial or GTI I.D. numbers of calibration kits, stopwatches, thermometers, bubble tubes, and pumps.
3. Measure the ambient air temperature with a mercury thermometer and obtain the water vapor pressure at the ambient temperature from appropriate tables. Obtain the barometric pressure from the NOAA radio station (frequency 162.55 MHz). Record all data.
4. Start the pump and check the battery condition by depressing the test button and observing the battery check LED.

ACCEPTABILITY: Battery Check LED should light

5. If the battery condition is unacceptable, place the pumps back on the charging rack and select another pump and repeat Step 4.
6. Place finger tightly over the pump inlet, wait 20-30 seconds and check the condition of the low flow indicator LED by depressing the test button.

ACCEPTABILITY: Low Flow Indicator LED should light

7. If results of Step 6 are unacceptable, mark the sampler with a tag indicating "INOPERATIVE-DO NOT USE", make an entry in the sampler's Instrument and Equipment History File (Q.C. Form 80-3). Remove the pump from service.
8. Record acceptable results from Steps 4 and 6 on the calibration sheet.
9. Allow the pump flow rate to stabilize and attach to the calibration system shown on the following page.

FLOW RATE CALIBRATION (CONT.)

10. Open Pressure Drop Valve so that Pressure Drop Meter reads zero (o).
11. Allow pump to run for 30 seconds for flow stabilization.
12. Use trial and error to establish target flow of 1270 cc/minute. Turn flow adjust control on pump to attain the desired flow rate and confirm with the bubble tube.
13. Once the flow rate has been achieved, confirm three (3) times using the bubble tube method. Record the calibration interval used on the bubble tube and the three (3) times for the bubble travel.
14. Calculate the Individual Flow Rates (STP) from the runs in Step 13 according to the following formula:

$$\underset{(\text{cc/min})}{\text{STP Flow Rate}} = \left[\frac{\text{Bubble Tube Vol. Used (cc)}}{\text{Travel Time (min)}}\right] \left[\frac{Pf - Pv}{Ps}\right] \left[\frac{Ts}{Tf}\right]$$

where Pf = atmospheric pressure (in. Hg) from NOAA 162.55 MHz (Step 3)
Pv = water vapor pressure at the calibration temperature (Step 3)
Ps = standard atmospheric pressure (29.92 in. Hg)
Ts = standard temperature (293°K)
Tf = ambient temperature of calibration (°K) (Step 3)

15. Calculate the Mean Flow Rate (STP) by summarizing the individual flow rates and dividing by three (3).

ACCEPTABILITY:
1) Mean Flow Rate (STP) must be within ±5% of the target 1270 cc/min flow rate (1206 to 1334 cc/min)
2) Individual Flow Rate (STP) (from Step 14) must be within ±5% of the Mean Flow Rate.

16. Pumps with flow rates not conforming to acceptability limits in Step 15 will be readjusted or removed from service.
17. Adjust the Flow Rate Valve and set the Flow Rate Meter to a predetermined value (approx. midscale). Record the Flow Rate Meter Reading.
18. Close the Pressure Drop Valve to give a reading of approximately 25 in. H_2O on the Pressure Drop Meter. Allow the pumpt to stabilize at the added pressure drop.
19. Record the flow rate as indicated on the Flow Rate Meter and compare to the flow calculated by the bubble tube method (Step 17).

ACCEPTABILITY: Flow Rate at 25 in. H_2O pressure drop must be within ±5% of Mean Flow Rate

20. Open Pressure Drop Valve so that Pressure Drop Meter reads zero and turn off pump.
21. Mark the pump with a colored sticker indicating that pump is calibrated and return to charging rack.

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-CP-009 Effective Date 082880

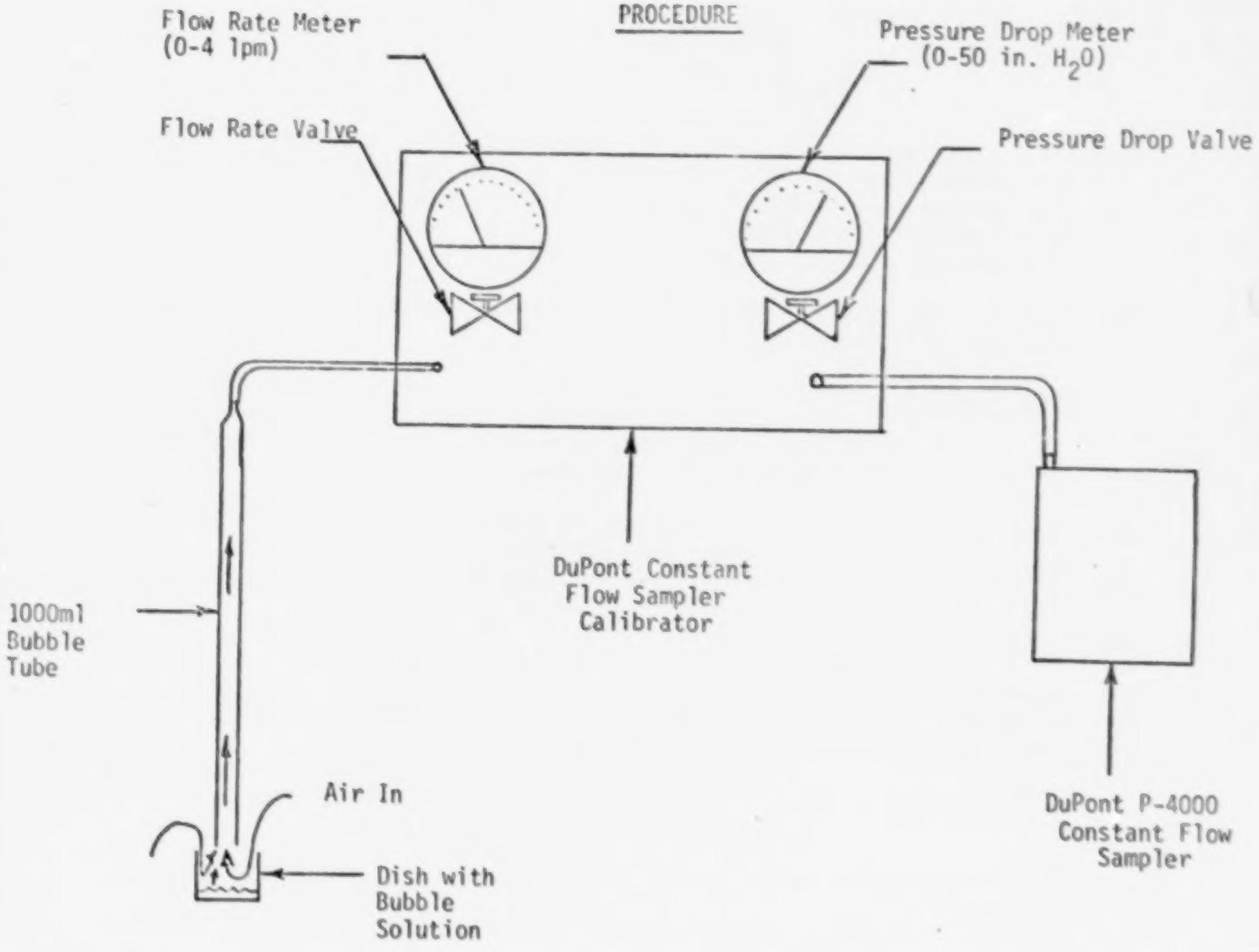
Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: DuPont P-4000 Constant Flow Sampler

Manufacturer: E.I. DuPont Model No: P-4000

Procedure Reference: P-4000 Operating Manual/GTI Staff

PROCEDURE


Flow Rate Meter (0-4 lpm)
Pressure Drop Meter (0-50 in. H_2O)
Flow Rate Valve
Pressure Drop Valve
1000ml Bubble Tube
DuPont Constant Flow Sampler Calibrator
DuPont P-4000 Constant Flow Sampler
Air In
Dish with Bubble Solution

CALIBRATION CONFIGURATION FOR DUPONT P-4000 SAMPLERS

Q.C. Form 80-7
Meteorology

GEOMET Technologies. Inc.

INSTRUMENT AND EQUIPMENT QC PROCEDURES LOG

Calibration

Proc. No.	Description	Date	Revision
GTI/LC-M-CP-001	Rain Gauge: Calibration	8/26/80	0
GTI/LC-M-CP-002	Wind Speed and Direction System: Wind Direction System Calibration	8/26/80	0
GTI/LC-M-CP-003	Wind Speed and Direction System: Wind Speed System Calibration	8/26/80	0
GTI/LC-M-CP-004	Hygrothermograph: Relative Humidity Calibration	8/26/80	0
GTI/LC-M-CP-005	Hygrothermograph: Temperature Sensor Calibration	8/26/80	0
GTI/LC-M-CP-006	Thermometers: Temperature Calibration	8/26/80	0

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-CP-001 Effective Date 082680

Procedure Type: Calibration X Operational Check ___ Maintenance ___

Instr./Equip. Item: Tipping-Bucket Rain Gauge

Manufacturer: Science Associates Model No: 562.563

Procedure Reference: GTI Staff

PROCEDURE

CALIBRATION OF RAIN GAUGE-GRAVIMETRIC METHOD

Step:	Description
1.	Determine the collection area of the rain bucket from the manufacturers literature or physical measurement and record.
2.	Calculate the volume of water required to produce a rainfall depth of one inch from the formula below:

$$\text{volume for 1-inch accumulation} = \underset{(\text{cm}^2)}{\text{collector area}} \times 2.54 \text{ cm}$$

3. To check the calibration, slowly introduce the volume of water (calculated in Step 2) into the collector. Record the volume of water used.
4. After the water has completely passed through the bucket assembly, check the reading on the digital accumulator and record.

ACCEPTABILITY: 1.00 ± 0.02 inches of water

5. Repeat Steps 3 and 4 two(2) more times.
6. If the accuracy in Step 4 cannot be achieved, the following items are checked and adjusted or repaired in the field:

   6a. bucket level
   6b. bad contact in gauge
   6c. electrical discontinuities
   6d. leak in bucket
   6e. drive or potentiometer

7. If adequate corrections cannot be made, the unit is replaced.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-CP-002 Effective Date 082680

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: Wind Speed Direction Measurement System

Manufacturer: Climatronics Model No: Wind Mark III

Procedure Reference: Instrument Manual/GTI staff f

PROCEDURE

WIND DIRECTION SYSTEM CALIBRATION

Step: Description

1. After installation of the system at each measurement site, establish a well documented true north location from examination of a USGS map selecting a landmark(s) or geographical feature(s) providing an accurate orientation. Record a detailed description of the items selected for orientation in a log notebook. Provide a photocopy of the map showing the features chosen for alignment for filing in the QC files.
2. Align the wind vane to indicate true north and adjust the transducer output to read 0°. Record the results on the data sheet.
3. Check the alignment of the system to the three (3) additional azimuth directions near 90°, 180°, and 270° by clamping the wind vane in place at each set direction and observing the transducer response on the strip chart. Record the results and label the strip chart accordingly.

ACCEPTABILITY: ±3° azimuth for each direction

4. In the event that the accuracy in Step 3 cannot be achieved, repairs or adjustments in system components will be made in the field. A notation describing the malfunction, subsequent repairs, and results of recalibration will be entered in the Instrument and Equipment History Record. If satisfactory performance cannot be achieved, the unit must be replaced.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-CP-003 Effective Date 0826800

Procedure Type: Calibration X Operational Check ___ Maintenance ___

Instr./Equip. Item: Wind Speed and Direction Measurement System

Manufacturer: Climatronics Model No: Wind Mark III

Procedure Reference: Manufacturer/GTI Staff

PROCEDURE

WIND SPEED SYSTEM CALIBRATION

Step:	Description
1.	The paired anemometer transmitter and cup assembly are calibrated by the manufacturer in a wind tunnel against a primary standard. A certificate of calibration received with the new instrument is filed as documentation of the calibration.
2.	At the end of the study when the system is dismantled, the unit will be returned to the manufacturer for recalibration. A certificate of calibration will be issued by the manufacturer upon recalibration. Any discrepancy between the initial and final calibration values for wind velocity accuracy will be taken into account in the data reduction and interpretation.

GEOMET TECHNOLOGIES. INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-CP-004 Effective Date 8/26/80

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: Hygrothermograph-Relative Humidity

Manufacturer: Casella (°C)/Weather Measure (°F) Model No: ____

Procedure Reference: GTI Staff

PROCEDURE

HYGROTHERMOGRAPH CALIBRATION FOR RELATIVE HUMIDITY

Step: Description

1. Use a sling psychrometer (an NBS-traceable thermometer) or an aspirated psychrometer as the reference standard for the relative humidity (RH) calibration. The thermometers on each unit (whether sling or aspirated) should be checked against an NBS traceable thermometer prior to use for this purpose.
2. Place Hygrothermograph in an environment that will not be subject to large fluctuations in temperature and humidity (i.e.. an air conditioned room) and allow at least 15 to 30 minutes equilibration. Verify that environment has been changed during this period with at least 3 psychrometer readings.
3. Upon equilibration. compare Hygrothermograph indication of relative humity to that derived from psychrometers.

ACCEPTABILITY: ±3% RH between relative humidity values of 20 and 80%

4. If the difference between readings exceeds control limit (±3% @ 20%≤ RH @80%). adjust relative humidity chassis screw (see manufacturers literature) and repeat Step 2 to verify that adjustment is proper. If this procedure does not remedy the deficiency. contact the meteorology supervisor before proceeding further.
5. If Step 4 corrects the problem. expose the unit to a new (and unchanging) environment (i.e.. outdoors in shade) and repeat Step 2. If the instrumental readings exceed control limits. repeat Step 2 in this environment before contacting meteorology supervisor or proceeding further.
6. If hygrothermograph continues to exceed control limits. the meteorology supervisor will decide whether to transport the faulty unit to GTI headquarters off site for recalibration of span or to seek replacement from either GTI resources or through purchase.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-CP-005 Effective Date 082680

Procedure Type: Calibration X Operational Check ___ Maintenance ___

Instr./Equip. Item: Hygrothermograph - Temperature

Manufacturer: Casella (°C); Weather Measure (°F) Model No: ___

Procedure Reference: GTI Staff

PROCEDURE

LABORATORY CALIBRATION OF HYGROTHERMOGRAPH TEMPERATURE SENSOR

Step: Description

1. Use mercury thermometers (ASTM 63F/ASTM 64F) traceable to an NBS standard test as the reference standard. Record the serial numbers of the thermometers used in each calibration.
2. Set up the hygrothermograph and reference thermometer on a table shielded from drafts or open doors.
3. Allow a minimum time of 30 minutes for the units to equilibrate to ambient temperature; record readings of both instruments during equilibration period (at least once every 10 minutes).
4. Record the hygrothermograph temperature reading and the temperature indicated on the reference thermometer.

ACCEPTABILITY: The hygrothermograph temperature should be within ±1.0°C (2.0°F) of the reference thermometer

5. If the acceptability limits in Step 4 are not achieved, adjust the hygrothermograph to read within the limits and repeat Steps 3 and 4 for verification. Record the results.
6. Place the hygrothermograph and reference thermometer in a similarly protected environment of different temperature and repeat Steps 2, 3, and 4. If response is unacceptable, repeat again before contacting meteorology supervisor.
7. If response continues to be unacceptable, the meteorology supervisor will decide whether to transport the faulty unit to GTI headquarters off site for recalibration of span or to seek replacement from either GTI resources or through purchase.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-CP-006 Effective Date 08/29/80

Procedure Type: Calibration X Operational Check ____ Maintenance ____

Instr./Equip. Item: Thermometers-ASTM 63F/64F; Psychrometer Thermometers

Manufacturer: Miscellaneous Model No: ____

Procedure Reference: GTI Staff

PROCEDURE

TEMPERATURE CALIBRATION

Step: Description

1. Use ASTM Thermometers 63F/64F (traceable to an NBS test) or thermometers calibrated against NBS-traceable thermometers as reference standards for this procedure.
2. Place the reference thermometers and the thermometers to be calibrated in a room or isolated box having a uniform temperature and allow the mercury to stabilize.
3. Read and record the temperatures indicated on the reference and test thermometers.

ACCEPTABILITY: 1. For Fahrenheit Thermometers. ±1.0°F
2. For Centigrade Thermometers. ±0.5°C

4. Select another area having a different temperature from that in Step 2 (i.e.. refrigerator. incubator. air conditioned room). Locate the thermometers near each other and allow the mercury columns to stabilize.
5. Proceed as in Step 3 using the same acceptability limits.
6. If the acceptability limits in Step 3 cannot be achieved. remove the thermometers from use and substitute alternate and equivalent units.

Q.C. Form No. [illegible]
Air

GEOMET Technologies. Inc.

INSTRUMENT AND EQUIPMENT QC PROCEDURES LOG

Operational Checks

Proc. No.	Description	Date	Revision
GTI/LC-A-OP-001	DuPont P-125 Air Samplers: Field Operational Check Procedures	8/28/80	0
GTI/LC-A-OP-002	DuPont P-4000 Air Samplers: Field Operational Check Procedures	8/28/80	0
GTI/LC-A-OP-003	High Volume Air Sampling Systems: Operational Checks	8/29/80	0

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-OP-001 Effective Date 082880

Procedure Type: Calibration ___ Operational Check X Maintenance ___

Instr./Equip. Item: DuPont P125 Constant Flow Sampler

Manufacturer: E.I. Dupont Model No: P-125

Procedure Reference: P-125 Operating Manual, GTI Staff

PROCEDURE

FIELD OPERATIONAL CHECK PROCEDURES

Step: Description

1. Select assigned pump for specific sampling location.
2. Remove pump cover and turn pump on. Observe to see that pump is operating.
3. After 10 to 20 seconds of operation, depress test button and observe the condition of the battery check LED.

ACCEPTABILITY: Battery check LED should light

4. If acceptability is indicated, check box on air sample data sheet.
5. If battery check LED does not light, select backup pump, and repeat Steps 2 and 3.
6. Depress test button and observe condition of low flow detector LED.

ACCEPTABILITY: Low Flow Detector LED should not light

7. Place finger tightly over pump air inlet and observe that pump motor speeds up (to compensate for increased resistance) after several seconds.

ACCEPTABILITY: Pump motor should increase in speed

8. After 20 to 30 seconds of operation with finger tightly over the pump inlet, depress the test button and observe the condition of the Low Flow Detector LED.

ACCEPTABILITY: Low Flow Detector LED should light

9. Release finger from pump inlet and observe the speed of the pump.

ACCEPTABILITY: Pump speed should return to normal

FIELD OPERATIONAL CHECK PROCEDURES (CONT.)

10. If checks in Steps 6 through 9 indicate acceptability, check feedback operational check box on air sampling data sheet, turn pump off, and install sampling media and pump on tripod.
11. If any check described in Steps 6 through 9 indicate unacceptable operation, use a backup pump and repeat Steps 2 through 10.
12. After media and pump are installed on tripod and all preparations are complete, turn pump on and check pump operation.

ACCEPTABILITY: Sampling pump motor should operate

13. After approximately 30 seconds of sampling, depress test button and check condition of low flow detector LED.

ACCEPTABILITY: Low Flow Detector LED should not light

14. If results of checks in Steps 12 and 13 indicate acceptability, sample collection may proceed.
15. If results of checks in Step 12 or 13 prove unacceptable, a new pump must be used and all operational checks must be performed.
16. Just prior to termination of sampling while sampler is operating depress test button and observe condition of low flow indicator LED.

ACCEPTABILITY: Low Flow Indicator LED should not light

17. If results of Step 16 indicate acceptability, check appropriate box on air sampling data sheet and terminate the sample.
18. If results of Step 16 are not acceptable (i.e., LED lights), make a notation indicating an uncertainty in flow rate on 1) the air sampling data sheet; 2) the Sample tag and; 3) the chain of custody sheet. Upon return to the GTI field office, the non-conformity should be brought to the attention of the supervisor in charge.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-OP-002 Effective Date 082880

Procedure Type: Calibration ____ Operational Check X Maintenance ____

Instr./Equip. Item: Dupont P-4000 Constant Flow Sampler

Manufacturer: E.I. Dupont Model No: P-4000

Procedure Reference: P-4000 Operating Manual, GTI Staff

PROCEDURE

FIELD OPERATIONAL CHECK PROCEDURE

Step: Description

1. Select assigned pump for specific sampling location.
2. Remove pump cover and check to see that all timer switches are bottom depressed in the OPEN position.
3. Turn the pump on and after 10-20 seconds of operation, depress the test button and check the condition of the battery check light LED.

ACCEPTABILITY: The Battery Check Light LED should light

4. If acceptability is indicated, check the battery operation box on the air sampling data sheet.
5. If the battery check light LED fails to light, select a backup pump and repeat checks in Steps 2 through 4.
6. Depress test button and observe condition of low flow detector LED.

ACCEPTABILITY: Low Flow Detector LED should not light

7. Place finger tightly over the pump air inlet and observe that pump motor speeds up (to compensate for resistance) after several seconds.

ACCEPTABILITY: Pump Motor should increase in speed

8. After 20-30 seconds of operation with finger tightly over the pump inlet, depress the test button and observe the condition of the low flow detector LED.

ACCEPTABILITY: Low Flow Detector LED should light

FIELD OPERATIONAL CHECK PROCEDURE (CONT.)

9. Release finger from pump inlet and observe the speed of the pump.

ACCEPTABILITY: Pump speed should return to normal

10. If checks in Steps 6 through 9 indicate acceptability, check feedback operational check box on air sampling data sheet, turn pump off, and install sampling media and pump on tripod.
11. If any check described in Steps 6 through 9 indicate an unacceptable condition, use a backup pump and repeat Steps 2 through 10.
12. After media and pumps are installed on tripod and all preparations are complete, turn pump on, record starting time, and check pump operation.

ACCEPTABILITY: Sampling pump motor should operate

13. After approximately 30 seconds of sampling, depress test button and check condition of low flow detector LED.

ACCEPTABILITY: Low Flow Detector LED should not light

14. If results of checks in Steps 12 and 13 indicate acceptability, sample collection may proceed.
15. If results of checks in Step 12 or 13 prove unacceptable, a new pump must be used and all operational checks must be performed.
16. Just prior to termination of sampling while sampler is operating depress test button and observe condition of low flow indicator LED.

ACCEPTABILITY: Low Flow Indicator LED should not light

17. If results of Step 16 indicate acceptability, check appropriate box on air sampling data sheet and terminate the sample.
18. If results of Step 16 are not acceptable (i.e., LED lights), make a notation indicating an uncertainty in flow rate on 1) the air sampling data sheet; 2) the Sample tag and; 3) the chain of custody sheet. Upon return to the GTI field office, the non-conformity should be brought to the attention of the supervisor in charge.

QC Form 80-4

GEOMET TECHNOLOGIES. INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-A-OP-009 Effective Date 8/29/80

Procedure Type: Calibration__ ____Operational Check X Maintenance____

Instr./Equip. Item: High Volume Air Sampling System

Manufacturer: Miscellaneous Model No:____

Procedure Reference: U.S. EPA (1977) Quality Control Procedures For Air Pollution Measurement Systems

PROCEDURE

HIGH VOLUME AIR SAMPLER OPERATIONAL CHECKS

Step: Description

1. After installation of high volume sampling system at each site. check Flow Recorder zero setting.

ACCEPTABILITY: Flow recorder should be set at zero while sampler is in "off" position

2. If the acceptability criteria in Step 1 is not achieved. adjust the recorder to the zero(0) position on the chart.
3. When sampling is initialed. allow the sampler to operate for approximately 5 minutes until the flow rate has stabilized. Check the flow recorder and record the initial flow.

ACCEPTABILITY: The stabilized initial flow rate should be within ±10% of the established initial flow rate (using a representative sample of filter media)

4. If the stabilized initial flow rate exceeds ±10% of the established flow rate. use a backup sampling unit.
5. After completion of sampling. check the flow recorder and determine the mean flow rate visually.

ACCEPTABILITY: The mean flow rate should be in the range of 30-60 C.F.M.

6. If the mean flow rate is outside of the range in Step 5. make notations on 1) air sampling data sheets. 2) sample tags. and 3) chain of custody forms.

HIGH VOLUME AIR SAMPLER OPERATIONAL CHECKS (CONT.)

7. Visually check the condition of the filter upon retrieval.

   ACCEPTABILITY: The filter should show a well-defined border around the particulate material. No evidence of leakage should be observed.

8. If the filter has a smudged border or evidence of leakage is apparent. make a notation on 1) air sampling data sheets. 2) sample tag. and 3) chain of custody forms.

Q.C. Form No. 80-8
Meteorology

GEOMET Technologies. Inc.

INSTRUMENT AND EQUIPMENT QC PROCEDURES LOG

Operational Checks

Proc. No.	Description	Date	Revision
GTI/LC-M-OP-001	Rain Gauge: Response Check	8/26/80	0
GTI/LC-M-OP-002	Wind Speed and Direction System: Operational Checks	8/26/80	0
GTI/LC-M-OP-003	Hygrothermograph: Operational Reasonability Checks	8/26/80	0
GTI/LC-M-OP-004	Hygrothermograph: Temperature and RH Operational Checks	8/26/80	0
GTI/LC-M-OP-005	Aspirated Psychrometer: Operational Checks	8/26/80	0

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-OP-001 Effective Date 082680

Procedure Type: Calibration ____ Operational Check X Maintenance ____

Instr./Equip. Item: Tipping-Bucket Rain Gauge

Manufacturer: Science Associates Model No: 562,563

Procedure Reference: GTI Staff

PROCEDURE

RAIN GAUGE RESPONSE

Step: Description

1. During precipitation events, the gauge is checked to verify proper performance.
2. Check the precipitation recorder/accumulator by visual observation to determine if the system is responding in reasonable accordance with the intensity of the rainfall event.

   ACCEPTABILITY: Reasonable correlation between recorder/accumulator response and the intensity of the rainfall occurring during the time of inspection

3. Record a notation of the inspection in the log notebook.
4. If the system fails to respond or the response appears to be unreasonable (based on the experience of the inspector), the system is immediately checked for the following:

   4a. leak in bucket
   4b. drive or potentometer
   4c. stuck bucket
   4d. bad gauge contact
   4e. electrical discontinuents
   4f. bucket level

5. If corrective measures fail to yield proper performance, the unit is replaced.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-OP-002 Effective Date 8/26/80

Procedure Type: Calibration ____ Operational Check X Maintenance ____

Instr./Equip. Item: Wind Speed and Direction Measurement System

Manufacturer: Climatronics Model No: Wind Mark III

Procedure Reference: GTI Staff

PROCEDURE

WIND SPEED AND DIRECTION SYSTEM OPERATIONAL CHECKS

Step: Description

1. The operational checks performed in the field are reasonability checks to verify gross instrument performance.
2. Observe the existing meteorological conditions at each site at the time of the check performance. Record the wind direction found as evidenced by these observations. Record the relative wind speed (static, slow, fast, etc.) in the log notebook.
3. Record the instrument responses (wind speed, direction) by examination of the strip chart recordings (make the specific notations in the log notebook and on chart).

ACCEPTABILITY: The instrument responses should correlate reasonably with the existing observed meteorological conditions

4. In the event that a gross disparity between observed and instrument recorded conditions is found (i.e., slow or static anemometer response for strong winds, etc.), the meteorology supervisor must be contacted and the cause of the problem determined.
5. GTI personnel will endeavor to perform all necessary repairs or adjustments at the site or in the field office.
6. If the corrective action taken is sufficiently severe to effect the calibration, the instrument will be recalibrated.
7. Corrected equipment conforming to established acceptability limits will be returned to active use.
8. Corrected equipment not conforming to established acceptability limits will remain out of use and, if necessary, returned to the manufacturer for repair. Alternate backup equipment will be substituted.
9. All malfunction, maintenance, and repair events must be documented on the individual Instrument and Equipment History Record for the particular item.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-OP-003 Effective Date 082680

Procedure Type: Calibration ____ Operational Check X Maintenance ____

Instr./Equip. Item: Hygrothermograph

Manufacturer: Casella (°C)/Weather Measure (°F) Model No: ____

Procedure Reference: GTI Staff

PROCEDURE

HYGROTHERMOGRAPH OPERATIONAL REASONABILITY CHECKS

Step: Description

1. Check the drum clock setting for proper time indication and adjust if necessary.
   ACCEPTABILITY: ±60 min. compared to Digital LCD Quartz Watch

2. If acceptable limits in Step 1 are exceeded, adjust the drum clock time to conform to proper time.
3. Check the trend in the temperature and relative humity records.

   ACCEPTABILITY: The temperature trend indicated should be reasonable

4. If trends observed in Step 3 are not reasonable within expectations, contact meteorology supervisor for guidance-whether to remove the unit from service for unscheduled calibration.
5. Check instrument for overall proper operation.

   ACCEPTABILITY: A continuous temperature and relative humidity trace should be observed

6. If discontinuities in the trace appear, inspect inking system and refill/replace as warranted.

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-OP-004 Effective Date 8/26/80

Procedure Type: Calibration _____ Operational Check X Maintenance _____

Instr./Equip. Item: Hygrothermograph-Temperature and Relative Humidity

Manufacturer: Casella (°C)/Weather Measure (°F) Model No: _____

Procedure Reference: GTI Staff

PROCEDURE

HYGROTHERMOGRAPH TEMPERATURE AND RH OPERATIONAL CHECKS

Step:	Description
1.	Use an aspirated psychrometer which has been checked against a reference thermometer (NBS-traceable) to calibrate the hygrometer temperature. Record the thermometer number used for each calibration.
2.	Place the psychrometer inside the louvered hygrothermograph shelter at the site and allow it to equilibrate for a minimum time of 3 minutes.
3.	Record the hygrothermograph readings and the temperature and relative humidity derived from the psychrometer.

ACCEPTABILITY: The hygrothermograph temperature should be within ±1.0°C (2.0°F) of dry bulb temperature, relative humidity ±3% of wet bulb/dry bulb derivation

Step:	Description
4.	If the deviation in temperature exceeds the acceptability limit in Step 3, repeat Steps 2 and 3.
5.	If the deviation in temperature and/or relative humidity exceeds the acceptability limits in Step 3, contact the meteorology superivosr for guidance in determining whether the deviations are due to instrument drift versus laps against environmental changes.
6.	Meteorology supervisor will advise whether to repeat Steps 2 and 3 or to return unit to onsite headquarters for unscheduled laboratory calibration.

QC Form 80-4

GEOMET TECHNOLOGIES. INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-M-OP-005 Effective Date 8/26/80

Procedure Type: Calibration __ Operational Check X Maintenance __

Instr./Equip. Item: Aspirated Psychrometers

Manufacturer: Bendix Model No: Psychron

Procedure Reference: GTI Staff

PROCEDURE

OPERATIONAL CHECK PROCEDURES

Step: Description

1. Check thermometers on each unit for signs of damage.

   ACCEPTABILITY: Thermometers should be intact with no mercury separations.

2. Check reservoir level for adequate water.

   ACCEPTABILITY: Water reservoir should contain sufficient distilled water.

3. Turn unit on and check for operation.

   ACCEPTABILITY: Fan should operate at reasonable rate.

4. If thermometers are damaged. replace.
5. Refill reservoirs on units where necessary.
6. If fan fails to operate. replace batteries.

GEOMET Technologies, Inc.

INSTRUMENT AND EQUIPMENT QC PROCEDURES LOG

Operational Checks

Q.C. Form No. 80-8

Proc. No.	Description	Date	Revision
GTI/LC-B-OP-001	Mammal Traps: Field Operational Checks	8/29/80	0

QC Form 80-4

GEOMET TECHNOLOGIES, INC.

INSTRUMENT AND EQUIPMENT QUALITY CONTROL PROCEDURE

QC Procedure No: GTI/LC-B-OP-001 Effective Date 082980

Procedure Type: Calibration ______ Operational Check X Maintenance ______

Instr./Equip. Item: Mammal Traps

Manufacturer: Victor; Have-A-Heart Model No: ______

Procedure Reference: GTI Staff

PROCEDURE

TRAP FIELD OPERATIONAL CHECKS

Step: Description

1. Tripping mechanisms for traps are checked in the field just prior to setting.
2. Arm the tripping mechanism of each trap. Trip the mechanism allowing the trap to close. Check the condition of gate closure.

   ACCEPTABILITY: 1. Tripping mechanism should operate properly.
   2. Gate closure should be secure.

3. If the trap appears to be malfunctioning, adjust the tripping mechanism. If adjustment is not effective in resolving the problem, use an alternate trap.

4.0 SAMPLE ANALYSIS

The subcontractors involved in analyzing samples for the Love Canal Study are listed below. The suggested QA Plan outline provided to these subcontractors is presented on the following page of this section.

Subcontractor	Project Analytical Work
Battelle Columbus Laboratories	Volatile organics on Tenax
PEDCO Environmental, Inc.	Volatile organics on Tenax
Gulf South Research Institute	Pesticides on polyurethane foam, organics in water, soil and sediments
Southwest Research Institute	Pesticides on polyurethane foam; soil, and sediments methods study; organics in soil, sediments, and biota; inorganics in water, soil, sediments, and biota.
Compuchem/Mead Technology Laboratories	Organics in water, soil, and sediments
PJB/Jacobs Engineering Group, Inc.	Organics in water, soil, and sediments; Inorganics in water, soil, and sediments
TRW, Inc.	Organics in water
Acurex Corporation	Soil and sediments methods study; organics in water, soil, and sediments
Energy Resources Company, Inc. (ERCO)	Inorganics in water, soil, and sediments
Midwest Research Institute	Organics in biota
Wright State University	Dioxin in all media
Advanced Environmental Systems, Inc.	TOC/TOX, pH, conductivity in groundwaters
Wadley Institutes of Molecular Medicine	Interferon in mice

SUBCONTRACTOR'S QA PROJECT PLANS FOR ANALYSIS OF SAMPLES FROM LOVE CANAL

Outline

1.0 Project Description and Organization

2.0 Major Project Elements

3.0 QA Objectives - State expected precision and accuracy to be achieved by each analytical method in each media analyzed.

4.0 Document Control Procedures - Overall framework, numbering system, sample tags, Custody Forms, Log Books, Data sheets provided by Prime Contractor. State your internal operation of the system, identifying responsible person as Document Control Officer.

5.0 Chain of Custody - Same as 4.0.

6.0 Analytical Internal QC Checks - Provide a table for each media (air, water, etc.) analyzed which lists operational checks for each analytical method. Control limits for each operational check should be provided with the corrective action to be taken if control limits are exceeded.

7.0 Data Reporting Management System - Standardized data forms will be provided by Prime Contractor - both analytical data sheets and encoding forms for the data management system. Internal operation of the system, and data review before reporting should be described.

8.0 Corrective Action Procedures - State how corrective actions will be implemented in the laboratory - identify responsible personnel. The corrective actions should have been identified in Section 6.0.

Appendix B Analytical Procedures

Part 1. Analytical Methods. Provided by Prime Contractor.

Includes analytical data sheets. Calibration of sampling equipment forms part of operating procedure--subcontractor provides for his equipment, unless given in analytical method.

Part 2. Analytical Equipment Maintenance/Troubleshooting Procedures

GC/MS

Analytical Balances

AA--Cold Vapor Hg, Furnace Techniques

pH and Selective Ion Meters

Technicon Autoanalyzer, If Used

Other Equipment

Appendix Q
Plan 4.1

Battelle Columbus Laboratories
QA Plan

QA PLAN FOR PROJECT ON ANALYSIS OF SAMPLES FROM LOVE CANAL

1.0 Project Description and Organization

This project is to determine organics in ambient air samples collected on Tenax -- to be supplied by the Prime Contractor.

The project organization is shown in Figure 1.

2.0 Major project elements are maintenance, GC/MS operation, computer and data handling, and internal QC as indicated in the Project Organization.

3.0 Objectives

3.1 The objectives of this QA plan are to provide analysis data of known quality and to be able to defend the quality of that data.

3.2 The expected precision and accuracy to be achieved for these analyses is 25% relative standard deviation and 25% relative error respectively.

Relative standard deviation and relative error will be estimated from replicate analyses of standards supplied by the contractor. Precision will be estimated from ranges of results on replicated standards. Relative error will be estimated from fit of data from a standards working curve. We will utilize both contractor supplied standards and internally prepared standards. The relationship to be used is

$$\frac{\text{Predicted - True}}{\text{True}} \times 100$$

where predicted is obtained from the inversion of the regression equation.

4.0 Document Control Procedures

4.1 The documents foreseen for this program include:

Laboratory Record Books
Chain of Custody Records
Procedures Description
Data Sheets

Laboratory Record Books (LRB) will be used to record all program activities. These are standard books used in all research at

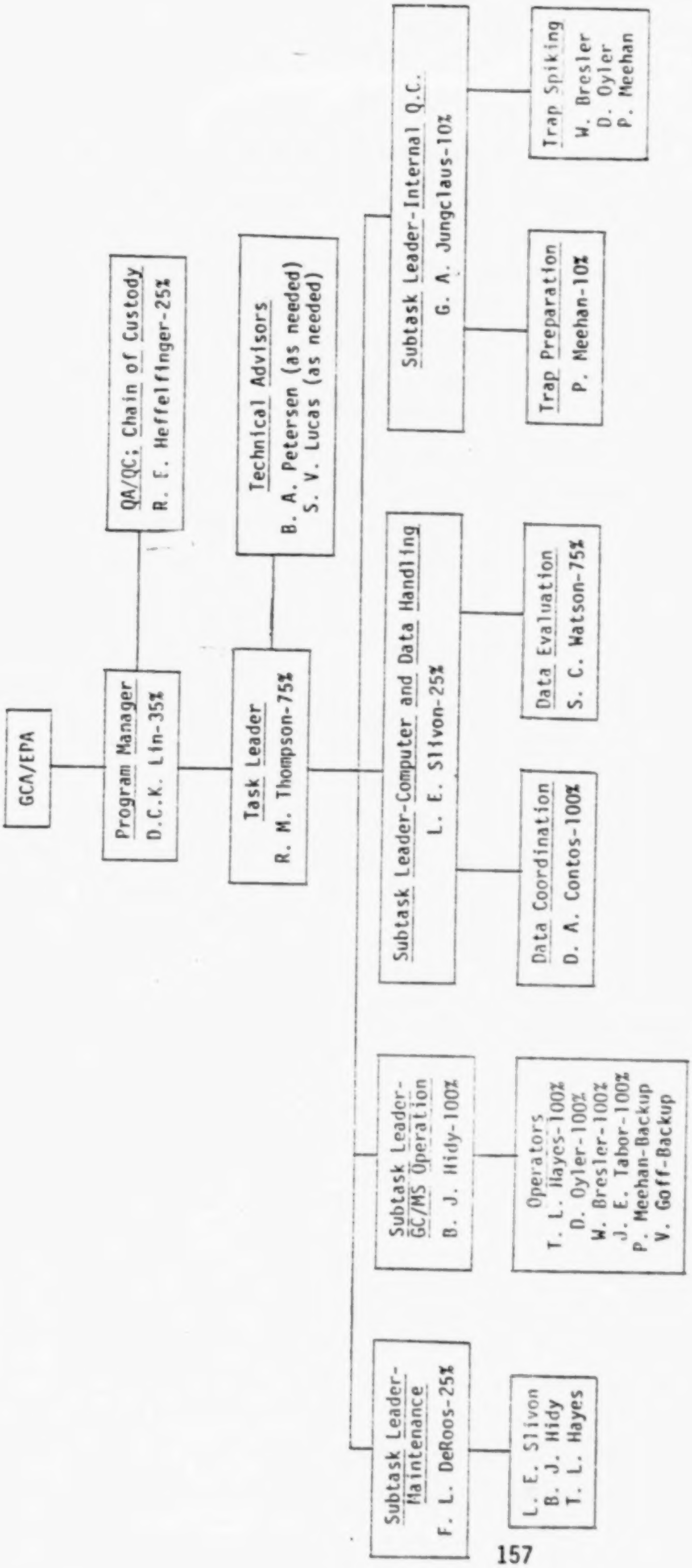

GCA/EPA
Program Manager
D.C.K. Lin-35%
QA/QC; Chain of Custody
R. F. Heffelfinger-25%
Task Leader
R. M. Thompson-75%
Technical Advisors
B. A. Petersen (as needed)
S. V. Lucas (as needed)
Subtask Leader-Maintenance
F. L. DeRoos-25%
L. E. Slivon
B. J. Hidy
T. L. Hayes
Subtask Leader-GC/MS Operation
B. J. Hidy-100%
Operators
T. L. Hayes-100%
D. Oyler-100%
W. Bresler-100%
J. E. Tabor-100%
P. Meehan-Backup
V. Goff-Backup
Subtask Leader-Computer and Data Handling
L. E. Slivon-25%
Data Coordination
D. A. Contos-100%
Data Evaluation
S. C. Watson-75%
Subtask Leader-Internal Q.C.
G. A. Jungclaus-10%
Trap Preparation
P. Meehan-10%
Trap Spiking
W. Bresler
D. Oyler
P. Meehan

FIGURE 1. PROJECT ORGANIZATION

Battelle and are numbered sequentially. We plan to use these books for the Chain of Custody Log, for the experimental log and for the QC sample log.

For the Chain of Custody log, information on each sample will be recorded to include all pertinent information (person delivering sample, person receiving sample, date and time received, source of sample, sample identification number, how transmitted, condition when received, analyst receiving sample, date of receipt, and date analysis is complete).

The book, page and line number where such information is recorded will establish the unique laboratory sample number for the sample described. A typical sample number will appear as: 32756-16-07.

The experimental log will contain sample identification information, information on instrumental performance check, and all information on experimental conditions. The data produced on hard copy will contain an information header that includes Lab sample number, user sample number, project number, date and time as well as the instrumental conditions and analysts name. These hard copies will be permanently bound and identified with the associated LRB. Through this system, complete traceability will be maintained.

The QC sample log will include all information on internally generated QC samples. Included in this log will be the kind of QC sample (blank, spike), spike concentrations, source and purity of spiking compounds, and reference to how the spike was made.

Chain of Custody records will include the Chain of Custody Log and all Chain of Custody sheets and tags. These will be filed in a locked cabinet.

Procedures descriptions. The procedures for analysis, for performance checks, for calibrations, and for quality control sample generation will be described. These descriptions are identified by numbers, revision number, and revision date. These identifying numbers will be used as references for we of the procedures in all experimental efforts.

Copies of the method descriptions will be in the laboratory and on file in the QA office.

Data Sheets. Copies of Analysis Data Reporting Forms will be copied for archival storage in project files and in the QA file. These sheets will carry identifiers as requested on the form as well as LRB numbers where raw data can be found.

All other documents such as memos and letters will contain a serialized number and will be subject to inventory.

5.0 Chain of Custody

In coming samples will be inventoried, the Custody Forms signed, and the samples stored in a locked cabinet kept at a maximum temperature of 4°C. The sample information will be logged and the unique laboratory number generated (see Section 4.0).

The custodian will distribute samples to the laboratory; the person receiving samples will sign and date the sample log book to that effect.

When the analyses are complete the samples will be stored at a maximum temperature of 4°C. The analysis completion date will be recorded in the sample record book.

6.0 The daily schedule for GC/MS instrument calibration and tuning, capillary column performance testing, quality control sample analysis, and actual sample analysis is as follows:

- Tuning GC/MS with bromofluorobenzene (BFB) via a batch inlet
- Calibration with FC-43 via batch inlet
- Direct injection of 2 µl of the standard mixture containing the 25 volatiles to be identified and quantified, BFB 10 ng/µl, and the six standards comprising the "Grob" mix. The concentration of each standard will be 25 ng/µl except for o-chlorotoluene, 1,2-dibromoethane, p-chlorobenzaldehyde, and 1,2-dichloroethane. The concentration of the latter four compounds will be 2.5 ng/µl. These four compounds will serve to evaluate the sensitivity of the system.
- Analysis of a GCA supplied standard QA cartridge of known concentration.

- Analysis of three actual samples
- Analysis of a GCA supplied blank cartridge
- Analysis of three actual samples
- Confirmation of GC/MS tuning with BFB via the batch inlet
- Analysis of GCA supplied standard QA cartridge of known concentration
- Analysis of three actual samples
- Analysis of GCA supplied blank cartridge
- Analysis of three actual samples
- Analysis of GCA supplied QA cartridge

The total daily operation from calibration through analysis of the final sample will require 18 hours.

Corrective Actions

Problem	Action
• Cannot tune mass spectrometer with BFB; ion ratios not acceptable.	1. Retune until ratios are correct.
• Cannot calibrate	1. Retune. 2. Check quadrupole rods. 3. Clean ion source and rods.
• Irreproducibility of retention times, resolution, search routines, and other capillary column parameters.	1. Check GC/MS operating parameters. 2. Check for leaks (fittings, septa, broken column ends, transfer lines). 3. Rerun test mixture by direct injection. 4. Rerun std GCA cartridge. 5. Change column.
• Cannot reproduce peak areas from QA standards to within 25% of expected values.	1. Check operation of NuTech: temperature, flows, and interface. 2. Check all fittings, septa, interface lines. 3. Check GC/MS sensitivity by direct injection of standard mixture.

	4. Run standard QA cartridge. 5. Change capillary column. 6. Clean ion source, rods. 7. Run blank cartridge after any standard cartridge which yields selected peak areas which are too high (indicating contamination).
• Excessive material in blank	1. Check liquid nitrogen traps. 2. Clean NuTech. 3. Run another blank.

Both manufacturer-supplied and in-house generated troubleshooting procedures are available for use in repairing the mass spectrometer should one fail at any time during this program.

Control charts will be maintained on calibration standards and for known QC samples.

A control chart will be developed from performance of standards with respect to a regression line calculated from the standards. Data on concentrations of standards and instrumental responses will be used to calculate a regression line. The response then will be used to determine a predicted value (X_p) of concentration from the inversion of the regression. The relative difference between the predicted value (X_p) and standard value (X_t) will be calculated for all concentrations by the expression,

$$R = \frac{X_p - X_t}{X_t}$$

The upper control limit is given by UCL = 2.66 $\overline{R}$ and the upper warning limit is given by UWL = 1.77 $\overline{R}$ where $\overline{R}$ is the average relative difference, R. This system is in place and computerized with hard copy output. A hard copy control chart will be documented for each compound. Each additional set of standards will be used to determine in or out of control.

Internal QC samples will be composed of standards prepared at BCL and blanks. Data from these will be treated to form control charts as above.

Duplicate analyses of samples or standards are not possible for analysis of Tenax traps, and thus precision of the analytical system cannot be measured. However, we plan to measure the repeatability of standard samples and from that information establish a precision control chart with control limits and warning limits established by 3 times and 2 times, respectively, the estimated standard deviation of the instrumental response -- probably in terms of predicted concentration (X_p). Standard deviation will be estimated from ranges until 10 to 12 replicates standards have been run. Naturally, this exercise can measure only the combined qualities of standard sample generation and analytical performance.

7.0 Data Management System

All raw data will be entered in the Laboratory Record Book along with any formulae used in calculations and with a sample calculation. Any print-out or strip chart associated with data acquisition will be bound and numbered sequentially along with the date and Lab Record Book number and page where the experiment is described.

The raw data will be checked by another person, who will date and sign the page(s) checked. This raw data will be transfered to analytical data sheets. All information on the data sheet will be checked against the raw data to assure accuracy of data transfer.

8.0 Corrective Action Procedures

The recognition of an out-of-control situation will be the responsibility of the analyst, the task leader, or the QA coordinator. An out-of-control signal may come from performance on standards or blanks. In addition, doubt may arise because of recognition by the analyst of unexpected changes in functioning of the analytical system. In any case of out-of-control, a report will be made for the record that includes a statement of lack of proper performance, how the problem was corrected, and which if any samples must be suspect. This out-of-control report will be numbered sequentially and archived in the program file. The prime contractor will be apprised of the facts.

PEDCo Environmental, Inc.
QA Plan

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ADDENDUM	PEDCo Environmental Laboratory Quality Assurance Plan	

Recipients

Greg Fusaro
Dwight Hayes
William Jess
Tim Sander
Tom Wagner
Doris White
Mary Lou Atkinson, GCA
RoseMary Ellersick, GCA

INTRODUCTION

This Q.A. plan supplements the PEI Laboratory Quality Assurance Plan. It adds specific details pertaining to Project No. 4215 but does not alter the intent or general policies of the overall plan.

SECTION 1

PROJECT DESCRIPTION AND ORGANIZATION

PEDCo Environmental is currently under subcontract to GCA/Technology Division to analyze air samples (Tenax cartridges) for the volatile organic compounds listed in Table 1-1 using a Nu-Tech Model 320 thermal desorption unit and combined gas chromatography-mass spectrometry (GC/MS) on a Finnigan Model 4023. The project code is LC 1-619-026.

Figure 1-1 shows our management plan. Mr. Gregg Fusaro, Group Leader for the Organic Analysis Group, is Project Manager. He is responsible for overall scheduling, work assignments, budget, and review and delivery of all products. He will directly supervise all analytical work and may perform analyses as well. He has final responsibility for validating and reporting data.

Mr. Thomas J. Wagner is assigned as coordinator and manager of all quality assurance functions as developed for this project. His responsibilities include all areas of work. He will report directly through the Project Manager to the GCA Project Officer.

TABLE 1-1. LOVE CANAL AIR MONITORING LIST - VOLATILE ORGANICS COLLECTED IN TENAX

COMPOUNDS TO BE QUANTIFIED

1. Benzene
2. Carbon tetrachloride
3. Chlorobenzene
4. o-Chlorotoluene
5. p-Chlorotoluene
6. 1,2-Dibromoethane
7. o-Dichlorobenzene
8. p-Dichlorobenzene
9. 1,1,2,2-Tetrachloroethylene
10. Toluene

COMPOUNDS TO BE QUALITATIVELY IDENTIFIED IF PRESENT

1. Chloroform
2. 1,2-Dichloroethane
3. 2,4-Dichlorotoluene
4. o-Chlorobenzaldehyde
5. p-Chlorobenzaldehyde
6. Benzyl Chloride (a-chlorotoluene)
7. 1,1-Dichloroethane
8. 1,1-Dichloroethylene (vinylidene chloride)
9. 1,2-Dichloroethane (EDC)
10. 1,2-Dichloroethylene
11. Dichloromethane
12. Phenol
13. o-Xylene
14. m-Xylene
15. p-Xylene

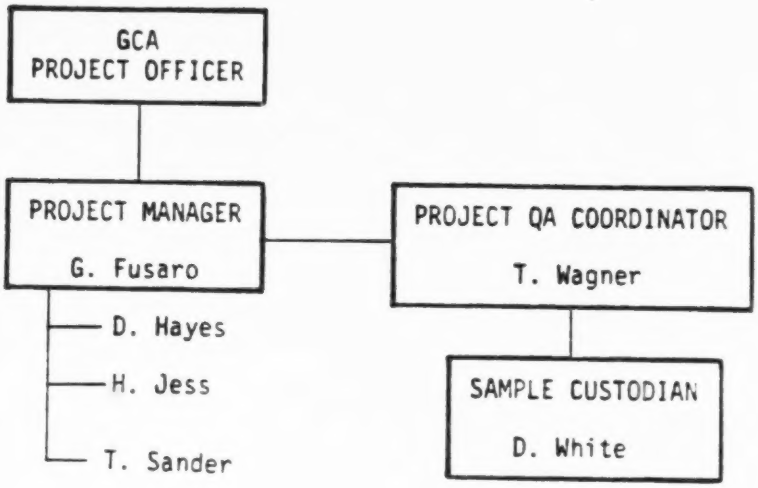

GCA
PROJECT OFFICER
PROJECT MANAGER
G. Fusaro
D. Hayes
H. Jess
T. Sander
PROJECT QA COORDINATOR
T. Wagner
SAMPLE CUSTODIAN
D. White

Figure 1-1 Project organizational chart.

SECTION 2

MAJOR PROJECT ELEMENTS

For this project only Tenax cartridges for the volatile organic compounds listed in Table 1-1 will be analyzed. The major quality assurance elements for these analyses are:

- precise and accurate analyses
- maintenance of analytical schedule and reporting schedule
- maintaining the Chain-of-Custody

The precision and accuracy of the analyses will be determined and maintained by the internal quality control (QC) checks described in Section 6 and external audit samples supplied by the prime contractor. The timeliness of the analyses and reports is the responsibility of the project manager; the sample custodian is responsible for maintaining chain-of-custody documentation.

SECTION 3

QUALITY ASSURANCE OBJECTIVES

The major Q.A. elements were listed in Section 2; the expected precision is 10% and the expected accuracy is ±20%. The analytical precision for each compound on the Tenax cartridges will be determined from the initial triplicate analysis of standards. The accuracy of the analyses will be checked every 8 hours of GC/MS time with one calibration standard that must fall within ±20% of the concentration determined from the calibration curve for each component. If this requirement is not met, appropriate corrective action listed in Section 6 is taken.

SECTION 4

DOCUMENT CONTROL PROCEDURES

4.1 GENERAL INFORMATION

The purpose of document control is to ensure that all project documents will be accounted for when the project is complete. Table 4-1 lists the principal items subject to document control with records maintained by the person listed as Document Control Officer (DCO) for that document. Table 4-2 gives a more complete listing of documents generated during the project. PEDCo's Document Control Officer is Mr. Thomas Wagner.

4.2 QA PROJECT PLAN

Each page will contain a heading on the upper right-hand corner that indicates Section Number, Revision Number, and Page Number of Total Pages in the section. The appropriate document numbering for subcontractor QA Plans is included with subcontract transmittal letter. When a revision to a QA Plan is made, the Table of Contents will be retyped to show the most current revision of each section. Both the revision and the revised Table of Contents will be distributed to all recipients of the original QA Plan. These recipients will be listed at the end of the QA Plan Table of Contents and in the QC Manager's records.

4.3 PROJECT NUMBERING SYSTEM

4.3.1 Project Code

The project code for the Love Canal Study is LC 1-619-026; this number must appear on sample identification tags, custody records, logbooks, data sheets, control charts, project memos and reports, document control logs, corrective action forms and logs, QA Plans, and all other project records.

4.3.2 Preserialized Documents

Sample collection and analytical tags, and custody records have preprinted serial numbers on them. It is not necessary that a tag number match a custody record number; it is necessary that each subcontractor DCO maintain records which account for all serially numbered items received. If tags or custody records are damaged, lost or destroyed before use for their intended purpose, the serial number of the item and its disposition must be recorded.

TABLE 4-1. DOCUMENT CONTROL REQUIREMENTS

Item	Issued by	Numbering	Interim responsibility	DCO
Sample Collection Tags	Sample Bank	Preserialized	Sampling Subcontractor	GCA Sample Bank
Custody Records	Sample Bank	Preserialized	See Section 5.0	See Section 5.0
Field Logbooks	Sample Bank	LC 1-619-026-	Sampling Subcontractor	Sampling Subcontractor
Field Data Sheets	Sample Bank	LC 1-619-026-	Sampling Subcontractor	GCA Sample Bank
Written Communication	Various People	Number and page of personal notebook where item is cited	Recipient	Subcontractor
Photos or Drawings	Originator		Originator	Subcontractor
Analytical Sample Tags	Sample Bank	Preserialized	Shipper	Analytical Subcontractor
Laboratory Notebooks	Subcontractor	LC 1-619-026-	Analytical Subcontractor	Analytical Subcontractor
Analytical Data Sheets	GCA	LC 1-619-026-	Analytical Subcontractor	Analytical Subcontractor
QA Project Plan	GCA	Section, Revision, Page Number of Total Pages	GCA QA Manager	GCA QA Manager
QA Project Plans	Subcontractors		Subcontractors	

TABLE 4-2. SUGGESTED DOCUMENT CODES

Document	Code Letter
Project Work Plans	A
Project Logbooks	B
Sampling Logbooks	C
Sampling Data Sheets	D1, D2 etc.
Sampling Coding Form	E
Laboratory Notebooks	G
Laboratory Data Sheets	H1, H2 etc.
Laboratory Coding Form	J1, J2 etc.
Sample Logs	L1, L2 etc.
Internal Memos	M
External Written Communication	N
Confidential Information	O
Photos, Maps, Drawings	P
QA Plan	Q
Reports	R
Final Report	FR
Miscellaneous	X

4.3.3 Other Documents

Sample identification tags and custody records are accounted for by their preserialized numbers. Other documents use the following numbering scheme to enable unique identification of each item:

Project Code	Subcontractor Code	Document Code	Serial Number
LC 1-619-026-	-21-	-A-	-00001

Table 4-2 presents a suggested document code. Each subcontractor's DCO will finalize the system to be used for his documents; it is desirable that consistent document codes be used by all subcontractors to facilitate compiling the overall project document inventory.

PEDCo ENVIRONMENTAL LABORATORY
SAMPLE RECEIPT AND RECORD SHEET

Sample Number	Requisition Number	Client Name	Client's Sample Identification & Type	Project Number	Date Received	Date Analyzed	Date Departed	Month Billed

FIGURE 5-1 Example of Sample Receipt and Record Sheet

The white (top) sheet of the requisition form is placed in the requisition binder; the pink sheet is placed with the samples and later with the raw data. The yellow sheet is given to the person requesting the analyses. In some cases, the yellow copy may accompany some of the samples if a batch includes both inorganic and organic analyses. A copy of the Sample Analysis Requisition and Record Sheet is shown as Figure 5-2.

The Tenax samples for this project will be stored at 4°C (40°F) in a refrigerator equipped with a high and low temperature alarm system. The samples remain in the locked sample storage room until removed for analysis. Transfer is documented on a Sample Control Record (Figure 5-3) which is maintained by the sample custodian. The Sample Control Record documents all custody changes which occur in the laboratory and each procedure performed on the sample. A set of Sample Control Records has been reserved for this project.

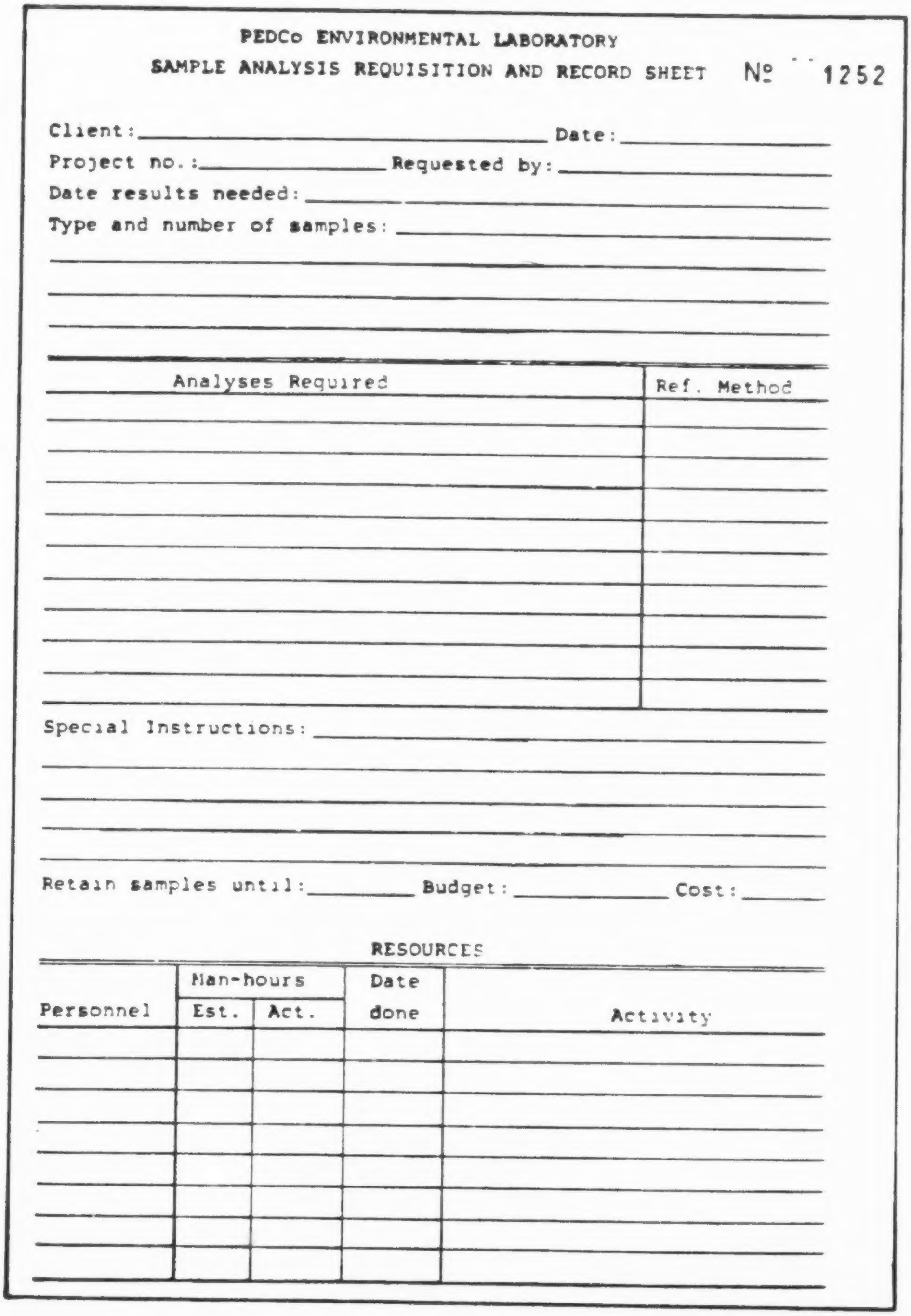
5.3 ANALYSIS

Release of samples requires annotation of the Sample Control Record and verification of information and sample container condition. If the sample is to be transferred between two persons (i.e., two analysts), the transfer must take place through the sample custodian. In other words, the sample must be returned to the sample custodian, and it is then reissued.

After obtaining the sample from the laboratory sample custodian, the analyst verifies the data and makes appropriate annotation of the records. The analyst keeps the samples in view or under limited-access locked storage. The analyst visually inspects the sample to determine that the physical condition is suitable for analysis. The analyst must maintain proper custodial procedures while analyzing a sample. Samples or intermediate solutions must be in the analyst's physical possession, in view, or in limited-access locked storage. The laboratories are locked so that only authorized personnel have access.

Analyses should be conducted in accordance with the procedures specified in the contract statement of work and referenced by number to the standard method in the laboratory procedures manual. Any deviation from these procedures must be annotated, and the analyst must be prepared to justify deviations under oath. All data are recorded on the data sheet. All measurements associated with the sample must be traceable in accordance with good quality assurance record-keeping procedures. Thus, associated calibrations must be recorded either directly on or attached to the data sheet or indirectly by reference to the standard solution number or instrument number.

Upon completion of the analysis the sample cartridges with sample tags attached are returned to the sample custodian.



PEDCo ENVIRONMENTAL LABORATORY

SAMPLE ANALYSIS REQUISITION AND RECORD SHEET № 1252

Client: ________________ Date: ________

Project no.: ________ Requested by: ________

Date results needed: ________________

Type and number of samples: ________________

Analyses Required	Ref. Method

Special Instructions: ________________

Retain samples until: ________ Budget: ________ Cost: ________

RESOURCES

	Man-hours		Date	
Personnel	Est.	Act.	done	Activity

FIGURE 5 -2 Example of Sample Analysis Requisition and Record Sheet

SAMPLE CONTROL RECORD

SAMPLE NO.	PEDCo NO.	DATE & TIME REMOVED	REASON	DATE & TIME RETURNED

FIGURE 5-3 Example of Sample Control Record

SECTION 6

ANALYTICAL INTERNAL QUALITY CONTROL CHECKS

6.1 INTRODUCTION

The routine quality control (QC) checks, their frequency, and criteria are listed in Table 6-1. The following gives more detail concerning these QC checks and the corrective action to be taken if the criteria are not met. Since these Q.C. checks are also specified in the analytical procedures, these procedures are included in Section 9.

6.2 INSTRUMENT TUNING

Perfluorotributylamine (BFTBA also known as FC-43) is used for mass calibration daily or every eight hours of instrument time whichever is more frequent. Bromofluorobenzene (BFB) is used for response calibration at the same frequency listed above. The criteria in Figure 6-1 must be met before any calibration standards or samples can be run. If the criteria cannot be met by tuning the instrument, the source is cleaned and the instrument retuned. If the criteria still cannot be met, professional service by the instrument manufacturer is obtained.

6.3 CAPILLARY COLUMN PERFORMANCE

Capillary column performance will be evaluated with "Grob" (acetophenone, 2,6-dimethylphenol, 2,6-Dimethylaniline, nonanone, ethyl benzene and para-xylene) mixture spiked onto calibration and quality control checking cartridges. The following criteria must be met to ensure adequate capillary column performance:

(1) $$\text{RESOLUTION} = \frac{2(W_1 - W_2)}{W_1 + W_2} = 1.20 \pm 11$$

where W_1 = Average base width of ethylbenzene measured at the base

W_2 = Average base width of p-xylene measured at the base

TABLE 6-1

COMPOUND	PURPOSE	FREQUENCY CODE	CRITERIA
PFTBA(FC-43)	Mass Calibration	1	Set mass markers
BFB	Response Calibration	1	Figure 6-1
Blank	Cross Contaminated	1 and after a heavily loaded sample >1000 ng of 1 component	25 ng*
Quantitation Standard	Quantitative Calibration	1	±20% of Calibration Curve

[1] daily or every 8 hours of instrument time whichever is more frequent.

* after a heavily loaded tube 300 ng because after a midpoint standard the blanks have been shown to be less than 25 ng.

Appendix Q
Plan 4.2

GC/MS PERFORMANCE STANDARD · BromoFluoroBenzene (BFB)

Date ______________ **Run no** ______________ **Analyst** ______________

Mass Spectrometer Performance Criteria

m/e	Ion Abundance Criteria			
50	15-40% of M/E 95			
75	30-60% of M/E 95			
95	Base Peak, 100% Relative Abundance			
96	5-9% of M/E 95			
173	Less than 1% of M/E 95			
174	>50% of M/E 95			
175	5-9% of M/E 174			
176	>50% of M/E 95			
177	5-9% of M/E 176			

Capillary Column Performance Criteria

Test Formula	Target Value	Act. Value
* Resolution = $\frac{2(W_1 - W_2)}{W_1 + W_2}$	≤ 1.20	
° % Peak Asymmetry Factor = $\frac{B}{F} \times 100$ Acetophenone	≤ 260	
° % Peak Asymmetry Factor = $\frac{B}{F} \times 100$ Nonanone	≤ 130	
Acidity test = $\frac{\text{Area of dimethylaniline}}{\text{Area of Acetophenone}}$	$= 1.0 \pm 0.1$	
Basicity test = $\frac{\text{Area of Dimethylphenol}}{\text{Area of Acetophenone}}$	$= 0.8 \pm 0.03$	

* W_1 = Base width of ethyl benzene
W_2 = Base width of P-xylene

° B = Area of back half of peak measured at a height of 1/10 of total peak height.
F = Area of front half of peak measured at a height of 1/10 of total peak height.

FIGURE 6-1

(2) PERCENT PEAK ASYMMETRY FACTOR (PAF) $\% \text{ PAF} = \frac{B}{F} \times 100$

where B = Area of back half of peak

F = Area of front half of peak

Both measured at 10 percent above baseline

% PAF for acetophenone = 260 + 34

% PAF for nonanone = 130 ± 32

(3) ACIDITY TEST (A.T.) = 1 ± 0.1 $\text{A.T.} = \frac{\text{PAWB}}{\text{PAA}}$

where PAWB = Peak area of dimethylaniline

PAA = Peak area of acetophenone

(4) BASICITY TEST (B.T.) = 0.8° ± 0.03 $\text{B.T.} = \frac{\text{PAWA}}{\text{PAA}}$

where PAWA = Peak area of dimethylphenol

PAA = Peak area of acetophenone

6.4 THERMAL DESORPTION SYSTEM

Proper operating temperatures and gas flow rates must be verified for at least each 8 hours of operation.

6.5 CALIBRATION

Before initiation of sample analyses a 3-point calibration curve in triplicate is to be generated using standard Tenax cartridges.

6.6 QUANTITATION CALIBRATION CHECKS

With each batch (nominally 42 tubes) of samples, 9 standard tubes should be run for quality control. This should correspond approximately to the analysis of one of the three concentrations of the calibration curve for each 8 hours of GC/MS time; this cartridge should be run during the first half of the 8-hour period. In any case, the controls should be spread evenly over the batch analysis period.

Corrective action must be taken if the observed concentration of any component, based on the original calibration curve, from the desorbing checking cartridge is greater than ±20 percent of the actual concentration. Corrective action could include retuning of the mass spectrometer, verification of capillary column performance, or verification of cleanliness of the system by running a blank cartridge. A second cartridge representing the same concentration can then be thermally desorbed and analyzed. If the observed concentration is still greater than ±20 percent of the actual concentration, based on the original calibration curve, a new calibration curve must be generated before any samples are analyzed.

6.7 BLANKS

In addition to the above routine quality control procedures, constant monitoring of the analyses for cross contamination must be established. Blanks will be analyzed at least daily or every eight hours of instrument time whichever is more frequent. In addition, a check of cross contamination will be made by analyzing a blank cartridge immediately after a heavily loaded sample has been analyzed. That is a sample which contains more than a 1000 ng. of any individual component. A careful record of the sequence in which these blank cartridges are run must be kept. The samples themselves are to be run in the sequence in which they are numbered when they are received from the Sample Bank at Niagara Falls.

SECTION 7

DATA REPORTING MANAGEMENT SYSTEM

7.1 IDENTIFICATION

The compounds listed in Table 1-1 are initially identified by a semi-automatic computer procedure which performs the following tasks:

A) The computer searches for the internal standard based on the retention time obtained for that compound from a standard run.

B) Once the internal standard is found, the computer searches in retention time windows determined by expected relative retention time to see if a particular compound is present. The three ions designated by EPA for each compound are displayed on the screen. If a response is observed the analyst examines it to be sure that the retention time is correct and that the three ions appear in approximately the correct ratios. If these conditions are met the analyst integrates the quantitation ion listed in Table 7-1.

C) This procedure is repeated for each compound until windows for all of the compounds have been examined.

D) Following this operation the computer calculates and prints out the concentration of each component using the response factors obtained from the standard curve.

Although the above computer procedure provides a preliminary identification of pollutants, the actual identification is performed by visual comparison of the sample mass spectra to those obtained from the standard run during the same shift. Both enhanced and unenhanced spectra are compared to see if the relative abundance, contamination, and retention time criteria are met. If all criteria are met, the compound is printed in the quantitation list.

TABLE 7-1

QUANTITATION IONS FOR SPECIFIED COMPOUNDS

COMPOUND	m/e	COMPOUND	m/e
Benzene	78	1,2-dibromoethane	107
Carbon tetrachloride	117	o-dichlorobenzene	146
Chlorobenzene	112	p-dichlorobenzene	146
o-chlorotoluene	126	1,1,2,2-tetrachloroethylene	166
p-chlorotoluene	126	Toluene	91

7.2 REPORTING

The verified data is given to the project manager who inspects the results and enters them on the Data Coding sheets supplied by the Prime Contractor. The report is reviewed by the project manager, before being issued. The report is to be issued no later than 30 days after receipt of the samples. A copy of the report is placed with the data in the project file.

In addition the data is transfered to 9-track magentic tape according to the following instructions; the tape is delivered within 35 days of sample receipt.

As the analyses are completed, GC/MS raw data is transferred to magentic tape as described below. All raw GC/MS data is to be retained on 9-track magnetic tape. When an analysis batch is completed, the tape is delivered to the Prime Contractor for storage and later evaluation. The tape format is:

Type - 9-track, 800 BPI, 2400 foot reels
Record length - 80
Block size - multiple of 80 (specify)
Code - ASCII

The data format must be approved by the Prime Contractor. At the present time there are formats used by commercially available equipment that may be acceptable. Before submitting tapes to the Prime Contractor, a sample data tape with appropriate documentation should be submitted for approval of the format and content.

Each GC/MS run on tape should contain the following header information at minimum.

1. Identifying run number
2. Data and time of sample run
3. Run description. Include GC column used, temp. program (e.g., 70-270-8), concentration factor of extract, run type (e.g., Base/Neutral, Acid, VOA, direct aqueous injection)
4. Scan time in seconds
5. Mass range scanned

Each scan within each run should contain at minimum:

1. Identifying run number
2. Scan number
3. Date
4. Time of scan (time from start of run)
5. Total intensity of scan (not normalized)
6. If intensities are normalized, the mass and raw intensity of the base peak for the scan. Intensities may be normalized or unnormalized. Normalized values must have a dynamic range of at least 1000; unnormalized values must reflect the actual absolute intensity value. Masses may be nominal integer values.

Each magnetic tape reel shall be accompanied by a Table of MS response ratios and a descriptor sheet that includes the following information for each run.

1. Prime Contractor sample number
2. Laboratory sample number
3. Fraction type (Base/Neutral, Acid, VOA, Blank, Standard Runs, etc.)
4. Specific source (Air, Water, Soil/Sediment)
5. Date received
6. Date extracted
7. Date analyzed

SECTION 8

CORRECTIVE ACTION PROCEDURES

Corrective action could be initiated at three levels:

- analytical level during routine Q.C. checks by the analysts
- quality assurance level by the Q.A. Coordinator
- Supervisory level by the project manager

The implementation of the corrective action depends on the nature of the problem. If it is a routine situation such as requiring retuning of the instrument, the analyst immediately follows the required procedure. If the normal procedure does not remedy the situation, the laboratory supervisor/project manager is notified and appropriate action is determined and taken.

If the situation is such that the appropriate action must be determined, the person identifying the problem notifies the laboratory supervisor/project manager who may determine the corrective action to be taken; or he may consult the Q.A. Coordinator and project officer to determine the appropriate action. The Q.A. coordinator must be consulted if the appropriate action involves a change in internal procedures, and the project officer must be consulted if the appropriate action alters externally supplied procedures.

SECTION 9

ANALYTICAL PROCEDURES FOR VOLATILE ORGANICS ON TENAX

SUMMARY OF METHOD

Ambient concentrations of selected volatile organic compounds are monitored by collection of integrated samples on Tenax sorbent cartridges, using an appropriate air sampling system, and subsequent analysis. The volatile organic constituents are determined by thermal desorption of the Tenax cartridges such that the desorbed organics are first cryogenically trapped and then flashed onto a capillary column for GC/MS analysis.

APPARATUS AND MATERIALS

1. Desorption Equipment
   - Thermal desorption and trapping apparatus (Nutech Model 320 Thermal Desorption System, or equivalent).
   - Mass flow meter (Tylon Model FC 260, or equivalent), calibrated for He in the 0-10 cc/minute range
   - Liquid nitrogen
   - Ultrahigh purity helium
   - Vacuum dewar
   - Bubble meter
2. Gas Chromatograph
   - Analytical gas chromatographic system with capillary inlet and oven temperature programming capability and amenable to interface with the above thermal desorption equipment.
   - Capillary column: 50 M Chrompack SE-30 thick phase, wide bore, WCOT, 0.5 mm I.D. (film thickness of 0.8 to 1.0 μm).

3. Mass Spectrometer

- Mass spectrometer interfaced to the above gas chromatograph and to a data system with capabilities conforming to the specifications given in Method 624 Part 4.9, page 69532, Federal Register, Monday, December 3, 1979.
- Magnetic tape storage capability
- NIH/EPA library
- Software with a minimum capability of real-time acquisition of data, data manipulation and library searching.

CALIBRATION AND PERFORMANCE CRITERIA

1. Instrument Tuning BFB

Mass spectrometer performance criteria are those set forth in Section 10, Method 624, (Federal Register, December 3, 1979, pp. 65932-69539). Performance specifications (BFB) must be met daily or for every 8 hours of analysis time and a permanent record of each performance check maintained.

2. Capillary Column Performance

Capillary column performance will be evaluated with "Grob" (acetophenone, 2,6-dimethylphenol, 2,6-dimethylaniline, nonanone, ethyl benzene and para-xylene) mixture spiked onto calibration and quality control checking cartridges. The following criteria must be met to ensure adequate capillary column performance:

(1) $$\text{RESOLUTION} = \frac{2(W_1 - W_2)}{W_1 + W_2} = 1.20 \pm 11$$

where W_1 = Average base width of ethylbenzene measured at the base

W_2 = Average base width of p-xylene measured at the base

(2) PERCENT PEAK ASYMMETRY FACTOR (PAF) $\% \text{ PAF} = \frac{B}{F} \times 100$

where B = Area of back half of peak

F = Area of front half of peak

Both measured at 10 percent above baseline

% PAF for acetophenone = 260 ± 34

% PAF for nonanone = 130 ± 32

(3) ACIDITY TEST (A.T.) = 1 ± 0.1 A.T. = $\frac{PAWB}{PAA}$

where PAWB = Peak area of dimethylaniline

PAA = Peak area of acetophenone

(4) BASICITY TEST (B.T.) = 0.8° ± 0.03 B.T. = $\frac{PAWA}{PAA}$

where PAWA = Peak area of dimethylphenol

PAA = Peak area of acetophenone

3. Thermal Desorption System

Proper operating temperatures and gas flow rates must be verified for at least each 8 hours of operation.

4. Calibration

Before initiation of sample analyses a 3-point calibration curve in triplicate is to be generated using standard Tenax cartridges.

D. ANALYTICAL PROCEDURES*

The Tenax cartridges are analyzed by thermal desorption at 250°C for 10 minutes in thermal desorption system. The desorbed organics are cryogenically trapped and subsequently flashed onto the specified glass capillary column. The effluent from the capillary will be directly introduced into the mass spectrometer for analysis. The thermal desorption system manufacturer's operating manual should be followed during this operation.

The following GC conditions are to be used for analysis:

Injector temperature 200°C

Column Oven Conditions: Initial temperature - 25°C, Hold, 5 minutes, Ramp - 4°C/min. to 130°C, 15°C/min. to 220°C, Hold, 2 minutes

Approximate flow: 2 cc/min.

---

* Exceptions noted in contract.

The mass spectrometer must be operated to accommodate the narrow peak widths produced by the capillary column. For this reason the following mass spectrometer conditions are to be used:

Source temperature: 250°C

Source pressure: $<4 \times 10^{-6}$ torr

Scan time: <1 sec.

Mass range: 35 to 400 amu

Electron energy: 70 eV

The concentrations (ng. per tube) of the compounds designated for quantitation are determined by comparing their responses to the calibration curve. The minimum quantifiable limit is determined to be 50 ng. per compound per tube. Each laboratory will define its limit of detection for each component to be identified as a signal to noise ratio of 4 to 1 and report this value in ng. per tube. Components present at concentrations between this minimum detection limit and the minimum quantifiable limit are to be reported as "trace." The compounds designated for qualitative analysis should be reported as "present" or "not detected" as appropriate. In addition, all spectra collected are to be archived on magnetic tape in the specified format.

E. QUALITY CONTROL PROCEDURES

With each batch (nominally 42 tubes) of samples 9 standard tubes should be run for quality control. This should correspond approximately to the analysis of one of the three concentrations of the calibration curve for each 8 hours of GC/MS time; this cartridge should be run during the first half of the 8-hour period. In any case, the controls should be spread evenly over the batch analysis period.

Corrective action must be taken if the observed concentration of any component, based on the original calibration curve, from the desorbing checking cartridge is greater than ±20 percent of the actual concentration. Corrective action could include retuning of the mass spectrometer, verification of capillary column performance, or verification of cleanliness of the system by running a blank cartridge. A second cartridge representing the same concentration can then be thermally desorbed and analyzed. If the observed concentration is still greater than ±20 percent of the actual concentration, based on the original calibration curve, a new calibration curve must be generated before any samples are analyzed.

In addition to the above routine quality control procedures, constant monitoring of the analyses for cross contamination must be established. This can be accomplished by analyzing blank Tenax cartridges provided as well as analyzing a blank cartridge immediately after a sample has been analyzed which is heavily loaded with organics. A careful record of the sequence in which these blank cartridges is run must be kept. The samples themselves are to be run in the sequence in which they are numbered when they are received from the Sample Bank at Niagara Falls.

F. SAMPLE HANDLING AND PRESERVATION

The samples are received as part of a chain-of-custody sequence which must be maintained by the analytical laboratory. The samples are to be kept at 4°C until actually analyzed. The cartridges should not be removed from the sealed culture tubes until the time of thermal desorption. The cartridges must not be touched with bare hands; cloth gloves must be worn or appropriate tongs used to handle the cartridges. All thermally desorbed cartridges are to be stored in their original culture tube container until notified of their disposition.

G. ANALYTICAL EQUIPMENT MAINTENANCE/TROUBLESHOOTING PROCEDURES

The gas-chromatograph-mass spectrometers are calibrated when used, for sensitivity and accurate mass assignment. If the required sensitivity cannot be obtained, prescribed maintenance procedures are initiated. If these do not enhance the instrument response to the level desired, professional service is obtained.

PEDCo Environmental, Inc. maintains Preventive Maintenance and Service Contracts with the manufacturers of all major instruments in use in the laboratory (Finnigan Corporation, Hewlett-Packard Company, Perkin-Elmer Corporation).

GC/MS PERFORMANCE STANDARD · BromoFluoroBenzene (BFB)

Date ____________ Run no ____________ Analyst ____________

Mass Spectrometer Performance Criteria

m/e	Ion Abundance Criteria			
50	15-40% of M/E 95			
75	30-60% of M/E 95			
95	Base Peak, 100% Relative Abundance			
96	5-9% of M/E 95			
173	Less than 1% of M/E 95			
174	>50% of M/E 95			
175	5-9% of M/E 174			
176	>50% of M/E 95			
177	5-9% of M/E 176			

Capillary Column Performance Criteria

Test	Formula	Target Value	Act. Value
* Resolution	$= \frac{2(W_1 - W_2)}{W_1 + W_2}$	≤ 1.20	
° % Peak Asymmetry Factor Acetophenone	$= \frac{B}{F} \times 100$	≤ 260	
° % Peak Asymmetry Factor Nonanone	$= \frac{B}{F} \times 100$	≤ 130	
Acidity test	$= \frac{\text{Area of dimethylaniline}}{\text{Area of Acetophenone}}$	= 1.0 ± 0.1	
Basicity test	$= \frac{\text{Area of Dimethylphenol}}{\text{Area of Acetophenone}}$	= 0.8 ± 0.03	

* W_1 = Base width of ethyl benzene
W_2 = Base width of P-xylene

° B = Area of back half of peak measured at a height of 1/10 of total peak height.
F = Area of front half of peak measured at a height of 1/10 of total peak height.

Gulf South Research Institute
QA Plan

List of Sections

Section 1.0 - PROJECT DESCRIPTION AND ORGANIZATION
Section 2.0 - MAJOR PROJECT ELEMENTS
Section 3.0 - QUALITY ASSURANCE OBJECTIVES
Section 4.0 - DOCUMENT CONTROL PROCEDURES
Section 5.0 - CHAIN OF CUSTODY
Section 6.0 - ANALYTICAL INTERNAL QUALITY CONTROL CHECKS
Section 7.0 - REPORTING MANAGEMENT SYSTEM
Section 8.0 - CORRECTIVE ACTION PROCEDURES

Appendix B - Analytical Procedures

1.0 PROJECT DESCRIPTION AND ORGANIZATION

1.1 GENERAL INFORMATION

Gulf South Research Institute (GSRI) is currently under contract to GCA/Technology Division to conduct analysis of volatile organics, semi-volatile organics and pesticides in air, water, soil and sediment samples collected in the Love Canal Area of Niagara Falls, New York.

1.2 PRINCIPAL INVESTIGATORS

Dr. R. A. Novak will serve as Principal Investigator with Dr. J. E. Gebhart as Co-principal Investigator on this program. As principal investigator Dr. Novak has overall responsibility for performance of the proposed program. He will function as the primary interface between the GSRI project team and GCA. In the execution of these duties, Dr. Novak in conjunction with Dr. Gebhart

--Have overall responsibility for meeting program objectives within budget and on schedule.

--Administer and supervise all requirements of the contract.

--Direct formulation of the work plan and initiate work plan revisions in accordance with the project officer's directions.

--Have responsibility for assuring that required staffing levels and technical expertise are provided to the program.

--Keep the project officer informed of all aspects of the program activity including expenditures, progress, problems, and recommended solutions.

--Work with the task leaders in planning and conducting review meetings.

--Are available to staff for action on any problem areas at all times.

--Are available to the task leaders for action on any problem requiring additional management or technical support.

--Keep management informed on all matters relating to the program.

--Review program reports, directives, work plans, progress reports, and financial reports prior to issue.

1.3 TASK LEADERS

Ms. Wilma Subra will serve as task leader for the sample preparation team. Dr. Novak will be task leader of the team of GC chemists. Mr. William Yauger will be task leader of the GC/MS effort.

The task leaders will be responsible for each task identified in the scope of work. In general, the task leaders will be responsible for organizing and directing the technical activities within their tasks and for reporting task progress to the project director. In execution of their duties, the task leaders

--Assist in the planning for each phase of their tasks and in defining objectives and activities.

--Have responsibility for responding to work plan revisions related to their tasks.

--Advise the principal investigator of progress, needs, and potential problems of their task.

--Direct the task activities and monitor performance of task members to insure that they are adhering to high technical standards and to the budget and task schedule.

--Maintain contact with the principal investigator in areas of technical concern.

1.4 QUALITY ASSURANCE

Mr. Charles McKibben is the Institute Quality-Assurance Coordinator.

Mr. McKibben will have overall responsibility for the following QA functions:

--Oversee QA function.

--Audit record keeping and documentation.

--Audit quality-control reporting and feedback.

--Audit records of calibration and traceability of standards.

--Oversee analytical quality control, including evaluation of interferences, recoveries, precision, reproducibility, and qualitative specificity.

--Audit data validation and analysis procedures.

2.0 MAJOR PROJECT ELEMENTS

2.1 AIR SAMPLES - PESTICIDES AND RELATED COMPOUNDS (RFP Task II)

GSRI shall analyze a number not to exceed 520 polyurethane foam plug samples, by the procedures provided by GCA for pesticides and related compounds. The samples will be delivered to GSRI as 13 batches of 30 to 50 plugs per batch. The schedule for delivery will be one batch every 8 days. For each batch received, GSRI shall complete the analyses within 30 days of receipt.

2.2 WATER SAMPLES - ORGANIC COMPOUNDS (RFP Task III)

GSRI shall analyze a number not to exceed 150 water samples by selected procedures provided by GCA. In completion of this work, GSRI shall

--Analyze a number not to exceed 150 sample fractions for volatile organic compounds by Method 624 (RFP Subtask 1).

--Analyze a number not to exceed 15 sample fractions for semi-volatiles by Method D for water, at the direction of the Technical Monitor (RFP Subtask 5).

--Analyze a number not to exceed 150 sample fractions for pesticides and related compounds by the Method for Pesticides in Water (RFP Subtask 6).

--Whenever acrolein and/or acrylonitrile is detected in the performance of the volatile organic analysis, or if directed by the Technical Monitor, quantify acrolein and/or acrylonitrile in accordance with the Acrolein and Acrylonitrile Quanitation Procedure. The number of such quantitations shall not exceed 35 (RFP Subtask 7).

--Whenever benzidine is detected in the performance of the semi-volatile analysis, or if directed by the Technical Monitor, quantify benzidine in accordance with the Benzidine Quantitation Procedure. The number of such quantitations shall not exceed 35 (RFP Subtask 8).

--Perform qualitative identifications by the GC/MS Qualitative Analysis Procedure on a number not to exceed 50 samples, as directed by the Technical Monitor (RFP Subtask 10).

The definition of a sample, as referred to above, is that collection of sample fractions necessary to complete the analyses specified above submitted to GSRI with unknown organic content. GCA will supply certain standardization and quality control materials required to perform the work according to the selected procedures; however, the standardization process and quality control measures shall be performed at GSRI's own expense.

The samples will be delivered in several shipments of between 25 and 50 samples each over the course of the contract term, but not to exceed a maximum of 50 in any 30-day period.

2.3 SOIL AND SEDIMENT SAMPLES - ORGANIC COMPOUNDS (RFP Task V)

GSRI shall analyze soil and sediment samples by selected procedures provided by GCA. In completion of this work, GSRI shall

--Analyze a number not to exceed 120 sample fractions for volatile organic compounds by Method 624 (RFP Subtask 1).

--Analyze a number not to exceed 50 sample fractions for pesticides and related compounds by the Pesticides in Soils Method (RFP Subtask 10).

--Analyze a number not to exceed 10 sample fractions for pesticides and related compounds by the Pesticides in Sediments Method (RFP Subtask 11).

--Whenever benzidine is detected in the performance of the semi-volatile analysis, or if directed to do so by the Technical Monitor, GSRI shall quantitate benzidine in accordance with the benzidine quantitation procedure. The number of such quantitations shall not exceed 15 (RFP Subtask 14).

The samples will be delivered in several batches of 15 to 30 samples per batch over the course of the contract term, but not to exceed a maximum of 30 samples in any 30-day period.

3.0 QUALITY ASSURANCE OBJECTIVES

3.1 GENERAL INFORMATION

The analytical laboratory is responsible for maintaining a continuing record of the data from all quality control checks and for using these data to make decisions on the acceptability of the analytical results as they are acquired. A more complete description of the quality control checks is provided in Section 6.0.

3.2 PRECISION AND ACCURACY

The analytical protocols (Appendix B) to be used for the Love Canal program contain detailed descriptions of the quality control measures to be employed during the course of the study. The routine analysis of replicate and spiked samples will provided precision and accuracy data for assessing the validity of the analytical results. GSRI's prestudy estimates of the expected precision and accuracy to be achieved by each analytical method in each media analyzed are presented below.

Method	Media			
	Air	Water	Soil	Sediment
		(percent recovery ± S.D.)		
Volatile organics	N.A.	90±15	80±30	80±30
Semivolatile organics	N.A.	65±25	Unknown*	Unknown*
Pesticides	Unknown**	80±15	80±15	80±15

*Method not selected.
**Data to be developed during the course of the program.
N.A. - Not Applicable to GSRI.

These estimates are derived from GSRI's prior experience with the methods supplied in Appendix B, data presented as part of the methods and presentations given at the EPA's Seminar for Analytical Methods for Priority Pollutants, Norfolk, Va., 1979 & 1980. The actual precision and accuracy data for the Love Canal samples will be derived through the implementation of the quality control procedures described in the methods.

4.0 DOCUMENT CONTROL PROCEDURES

4.1 GENERAL INFORMATION

The purpose of document control is to maintain complete documentation of each step of the analysis of samples and to account for all controlled documents at the completion of the project.

4.2 OVERALL FRAMEWORK

The document control program is performed under the supervision of the document control officer (DCO). The document control officer for the overall program is Maxine Latiolais. The control of documents originating at the New Orleans laboratory of GSRI is supervised by Kay Magyari. THE DCO is responsible for issuing, controlling and maintaining records of controlled documents. The overall scheme of document control within GSRI is presented in Figure 4-1.

4.3 NUMBERING SYSTEM

The numbering system to be used on all control documents is as follows:

Project Code	Subcontractor Code	Document Code	Serial Number
LC 1-619-026-	22	-A-	A0001

The document code used by GSRI is presented in Table 4-1. The serial number is a letter followed by four numerical digits. The letter designates the issuing site within GSRI (A - New Iberia, B - New Orleans).

4.4 SAMPLE TAGS

Samples as received from the Sample Bank of GCA will be tagged with the analysis tag shown in Figure 4-2.

4.5 CUSTODY FORMS

The samples received from the Sample Bank of GCA are accompanied by the Chain of Custody Record shown in Figure 4-3.

4.6. SAMPLE CONTROL FORMS AND DATA SHEETS

The serialized forms used for sample control by GSRI upon receipt, extraction, cleanup, instrument analysis, data compilation, and data reporting are presented in Figures 4-4 through 4-8.

The document control officer is responsible for issuing the designated serialized documents and accountability for said documents.

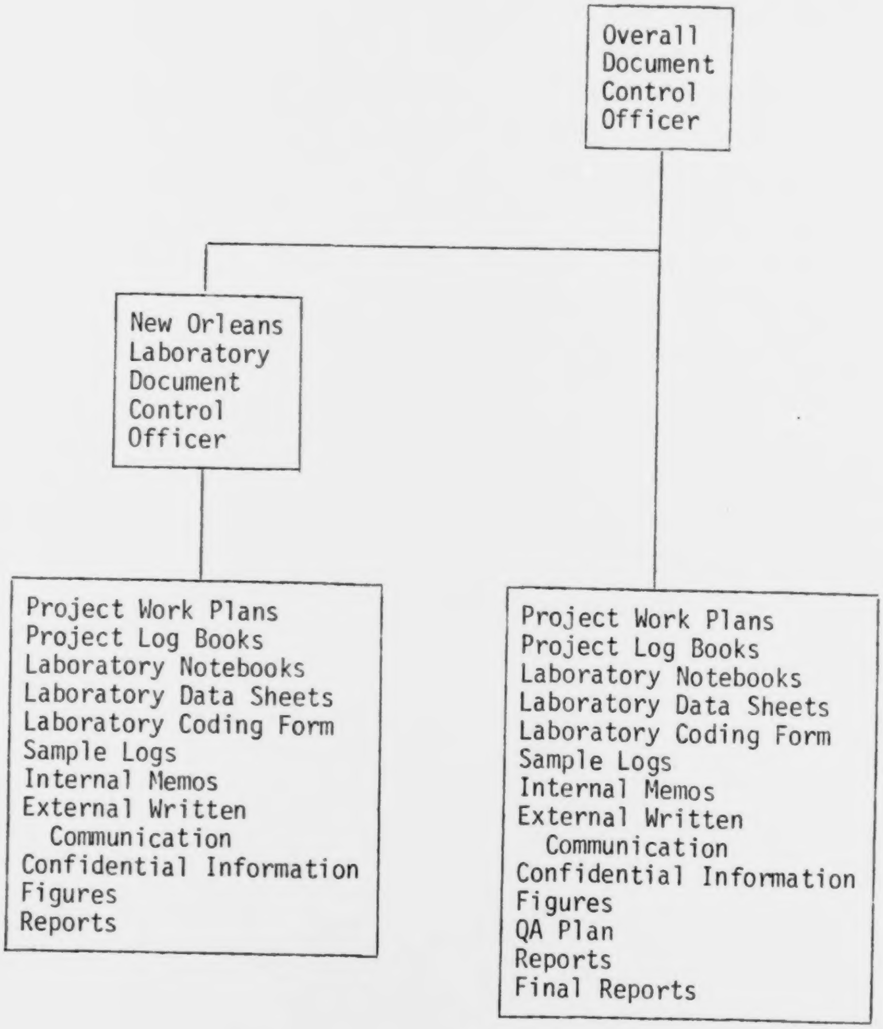
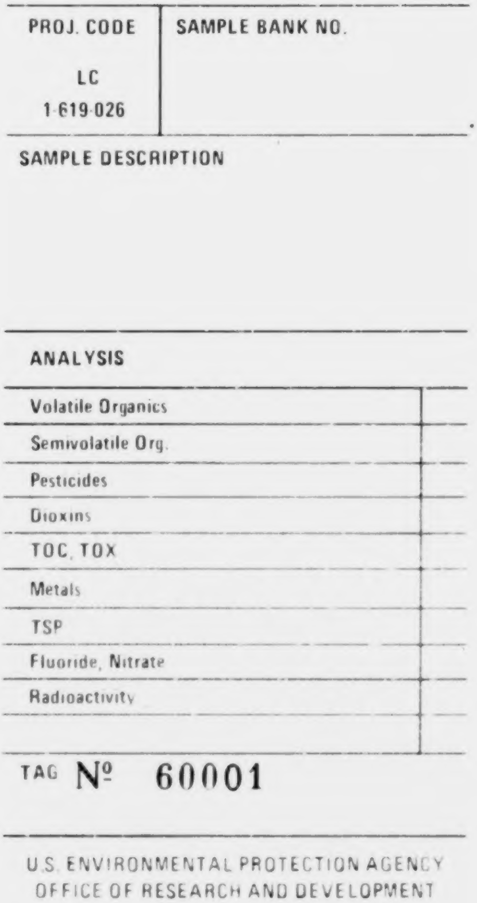

Overall Document Control Officer
New Orleans Laboratory Document Control Officer
Project Work Plans
Project Log Books
Laboratory Notebooks
Laboratory Data Sheets
Laboratory Coding Form
Sample Logs
Internal Memos
External Written Communication
Confidential Information
Figures
Reports
Project Work Plans
Project Log Books
Laboratory Notebooks
Laboratory Data Sheets
Laboratory Coding Form
Sample Logs
Internal Memos
External Written Communication
Confidential Information
Figures
QA Plan
Reports
Final Reports

Figure 4-1

Table 4-1. DOCUMENT CONTROL

Document	Code Letter
Project Work Plans	A
Project Log Books	B
Laboratory Notebooks	G
Laboratory Data Sheets	H
Laboratory Coding Form	J
Sample Logs	L
Internal Memos	M
External Written Communication	N
Confidential Information	O
Figures	P
QA Plan	Q
Reports	R
Final Reports	FR
Miscellaneous	X



PROJ. CODE LC 1-619-026	**SAMPLE BANK NO.**

SAMPLE DESCRIPTION

ANALYSIS

Volatile Organics	
Semivolatile Org.	
Pesticides	
Dioxins	
TOC, TOX	
Metals	
TSP	
Fluoride, Nitrate	
Radioactivity	

TAG № 60001

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT

Figure 4-2. GCA Sample Tag

Figure 4-3

ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development

CHAIN OF CUSTODY RECORD

Project Code LC	**Project Name** LOVE CANAL STUDY (1-619-026)	**ANALYSIS**
TO:	**SAMPLE DESCRIPTION**	

Sample Bank No	Tenax	Polyfoam	Hi-Vol	Water	Soil	Sediment	Other	Volatile Organics	Semivolatile Organics	Pesticides	Dioxins	TOC, TOX	Metals	TSP	Fluoride, Nitrate	Radioactivity	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks	

№ 2000 Distribution: Original Accompanies Shipment; Copy to Sample Bank Files

Figure 4-4

Sample Extraction Log

PROJECT SAMPLE NUMBER	GSRI SAMPLE NUMBER	EXTRACTION					CONCENTRATION				STORAGE LOCATION		COMMENTS
		Int Std Added	Solvent	Date	Person	Notebook Reference	Final Volume	Person	Date Ready for Analysis	Notebook Reference	Room Number	Refrigerator Number	

Figure 4-5

GC Analysis Log

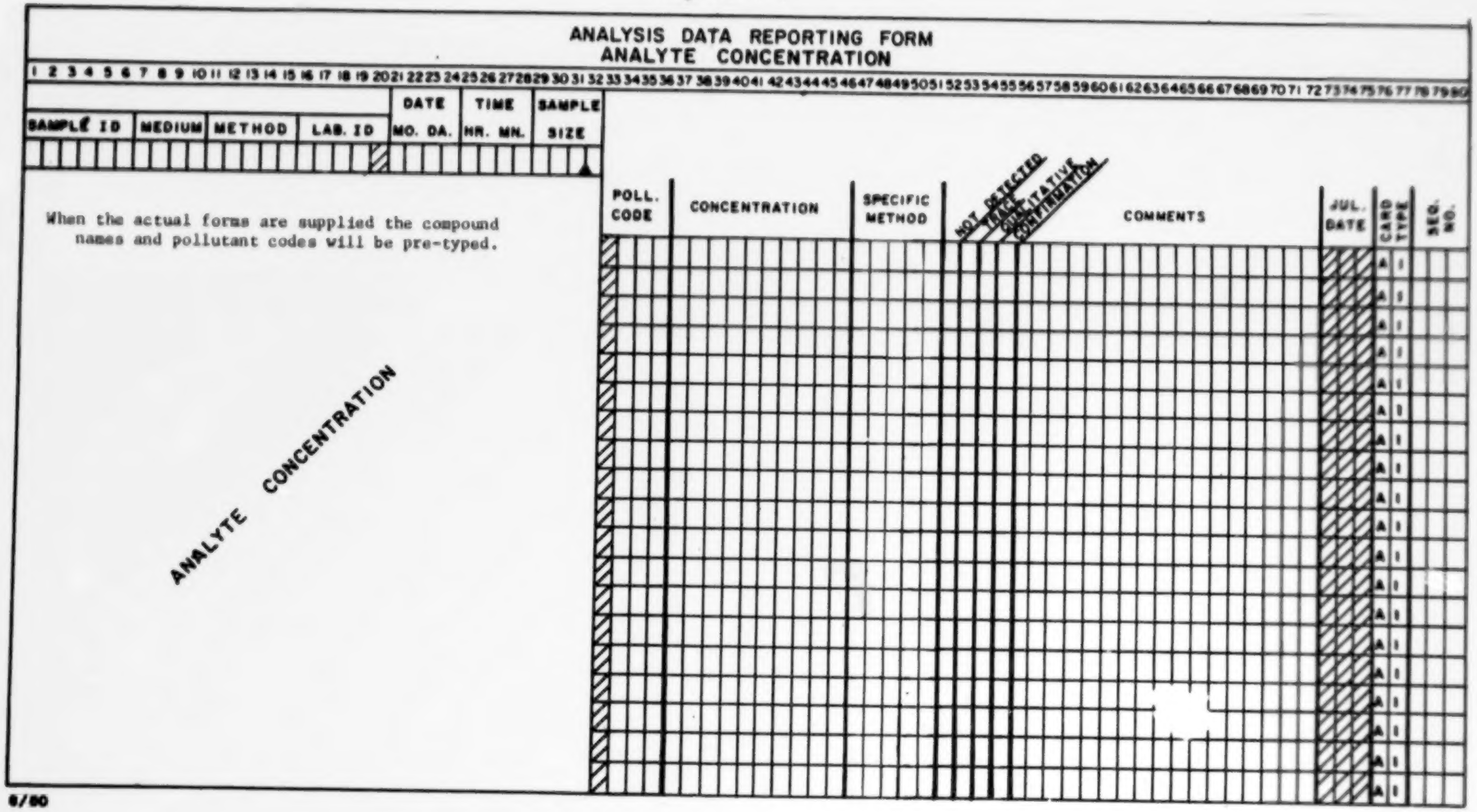
PROJECT SAMPLE NUMBER	CONDITIONS				ANALYSIS			STORAGE LOCATIONS		COMMENTS
	Instrument	Detector	Column	Temperature Program	Person	Date	Notebook Reference	Room Number	Refrigerator Number	

Figure 4-6

GC/MS Analysis Log

PROJECT SAMPLE NUMBER	CONDITIONS			ANALYSIS		STORAGE LOCATIONS		DATA REDUCTION		COMMENTS	TAPE TRANSFER		
	Instrument	Column	Temperature Program	Date	Person	Room Number	Refrigerator Number	Date	Person		Date	Person	Location

Figure 4-7



ANALYSIS DATA REPORTING FORM
ANALYTE CONCENTRATION

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80

SAMPLE ID	MEDIUM	METHOD	LAB. ID	DATE MO. DA.	TIME HR. MN.	SAMPLE SIZE

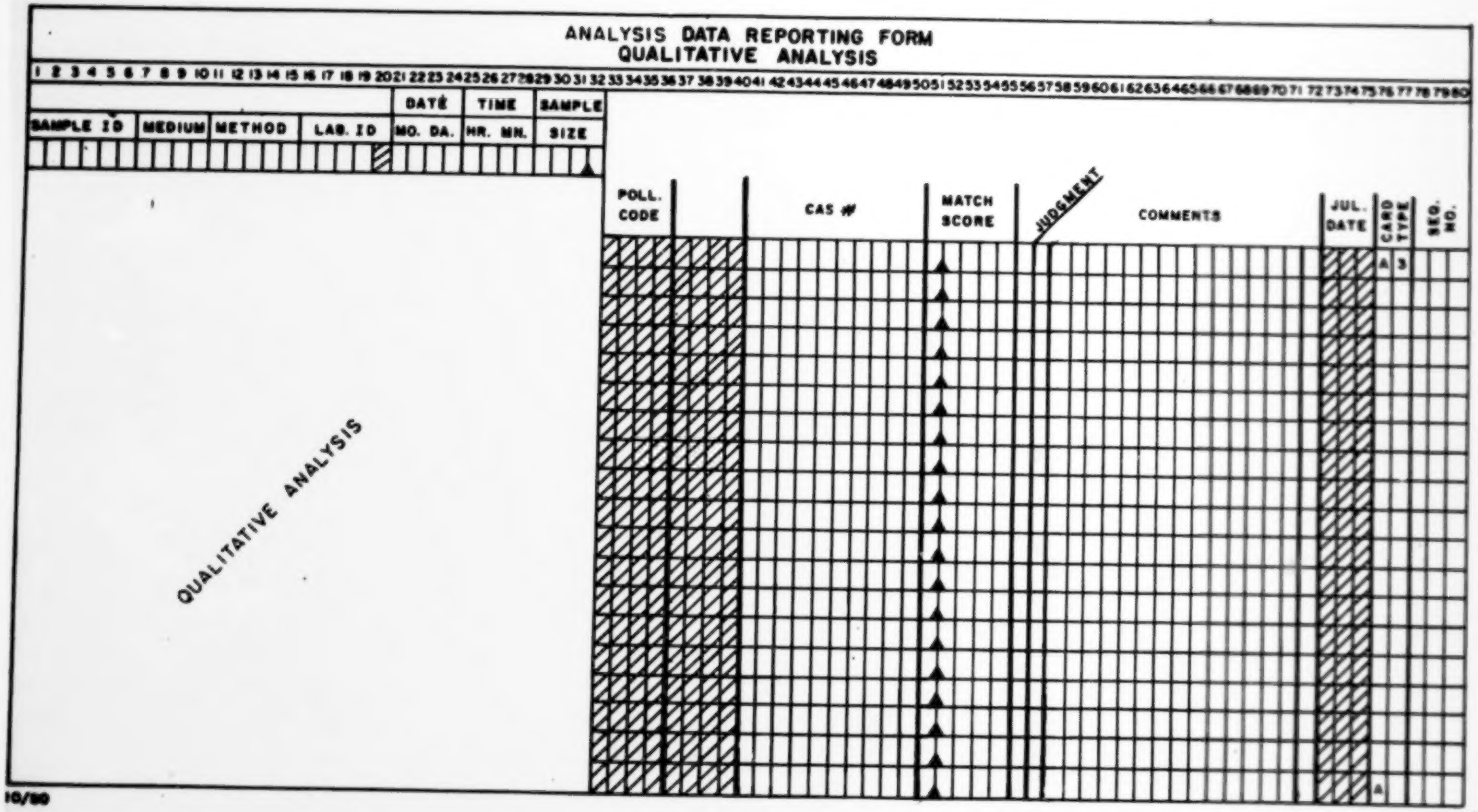
When the actual forms are supplied the compound names and pollutant codes will be pre-typed.

ANALYTE CONCENTRATION

POLL. CODE	CONCENTRATION	SPECIFIC METHOD	NOT DETECTED	TRACE	QUALITATIVE CONFIRMATION	COMMENTS	JUL. DATE	CARD TYPE	SEQ. NO.
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	
								A 1	

6/80

Figure 4-8



ANALYSIS DATA REPORTING FORM
QUALITATIVE ANALYSIS

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80

SAMPLE ID	MEDIUM	METHOD	LAB. ID	DATE MO. DA.	TIME HR. MN.	SAMPLE SIZE

POLL. CODE		CAS #	MATCH SCORE	JUDGMENT	COMMENTS	JUL. DATE	CARD TYPE	SEQ. NO.
							A 3	

QUALITATIVE ANALYSIS

10/80

4.7 LOG BOOKS

Serialized log books are issued by the DCO to individuals performing specific tasks on the project. All log book entries are dated and signed at the completion of the entry. Details of the handling of samples, problems, corrective action, in addition to information entered in sample control forms are entered in the log books. Sufficient detail is provided to enable others to reconstruct the analysis in the absence of the analyst.

5.0 CHAIN OF CUSTODY

5.1 GENERAL INFORMATION

The purpose of the chain of custody procedure is to document the transfer of custody of each sample from the sample bank of GCA to GSRI, through sample analysis, to analytical data reports.

5.2 OVERALL FRAMEWORK

The chain of custody is performed under the supervision of the sample custodian (SC). The SC for the overall program is Paul Conzelmann. The control of samples arriving at the New Orleans laboratory of GSRI is supervised by Lee Truxillo and the control of items arriving at the Contracts Office in Baton Rouge by Cindy Palazzo. The overall scheme of sample custody within GSRI is presented in Figure 5-1.

The sample custodian receives samples shipped to GSRI by the sample bank of GCA via Federal Express. Upon receipt of samples from Federal Express, the sample custodian documents the condition of the locked or strapped and sealed shipping box on the custody form (Figure 4-3). The custodian then checks the sample tag information against the custody record and notes the condition of the samples. Comments regarding the condition of the samples is noted under the remarks section of the custody form. Federal Express will then be requested to sign the form under "relinquished by" and the sample custodian, "received by".

The sample custodian logs the samples on the serialized form presented in Figure 5-2 and prepares sample set protocols to accompany each set of samples. The custodian then transfers the samples to the extraction chief. The extraction chief is responsible for the care and custody of the samples during the sample preparation phase of analysis. An internal log book will be kept to record all internal transfer of samples made by and to the sample custodian within the laboratory.

The extraction chief transfers the extracts to the sample custodian who verifies that the extracts correspond to the documents. The extracts are then transferred to the GC chemist (internal log book recording transfer) or to the GC/MS chemist (Chain of Custody Record filled out and executed) via Purolator Courier Service. The respective chemist will analyze the extracts, compile the data and transfer the complete data package to the sample custodian. Upon receipt of the complete data package, the sample custodian will verify the documents and transfer the data to the P.I.

Figure 5-1. Chain of Custody Within GSRI

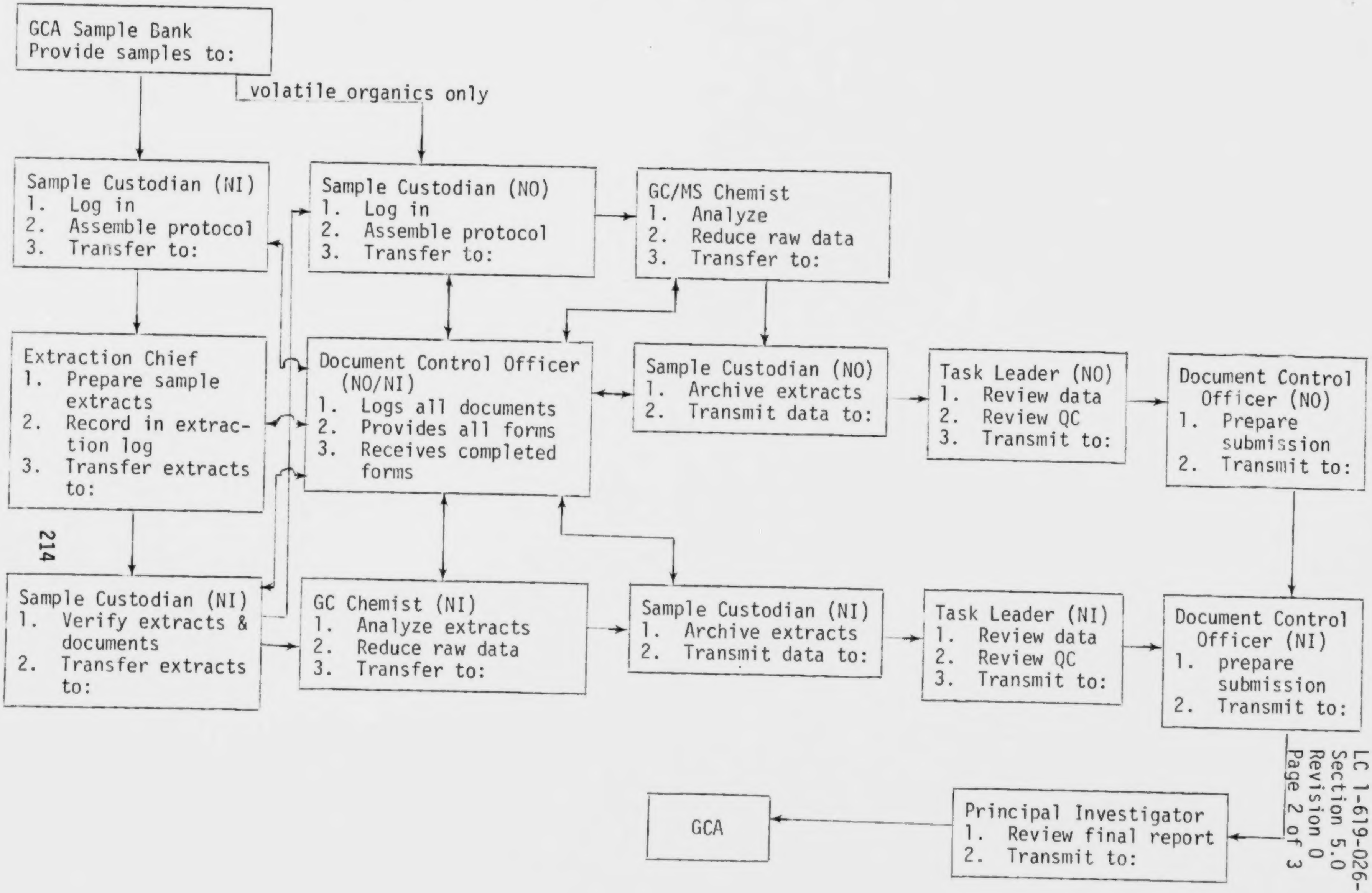

GCA Sample Bank
Provide samples to:
volatile organics only
Sample Custodian (NI)
1. Log in
2. Assemble protocol
3. Transfer to:
Sample Custodian (NO)
1. Log in
2. Assemble protocol
3. Transfer to:
GC/MS Chemist
1. Analyze
2. Reduce raw data
3. Transfer to:
Extraction Chief
1. Prepare sample extracts
2. Record in extraction log
3. Transfer extracts to:
Document Control Officer (NO/NI)
1. Logs all documents
2. Provides all forms
3. Receives completed forms
Sample Custodian (NO)
1. Archive extracts
2. Transmit data to:
Task Leader (NO)
1. Review data
2. Review QC
3. Transmit to:
Document Control Officer (NO)
1. Prepare submission
2. Transmit to:
Sample Custodian (NI)
1. Verify extracts & documents
2. Transfer extracts to:
GC Chemist (NI)
1. Analyze extracts
2. Reduce raw data
3. Transfer to:
Sample Custodian (NI)
1. Archive extracts
2. Transmit data to:
Task Leader (NI)
1. Review data
2. Review QC
3. Transmit to:
Document Control Officer (NI)
1. prepare submission
2. Transmit to:
Principal Investigator
1. Review final report
2. Transmit to:
GCA

Figure 5-2

GSRI Sample Custody Log

SPONSOR	SPONSOR SAMPLE NUMBER	SAMPLE TYPE	GSRI SAMPLE NUMBER	RECEIVING				PROJECT MANAGER	Date File Completed.
				Date	Person	STORAGE LOCATION			
						Room Number	Refrigerator Number		

6.0 ANALYTICAL INTERNAL QUALITY CONTROL CHECKS

6.1 GENERAL INFORMATION

This section describes the quality control procedures to be utilized throughout the analysis phase of the environmental survey of the Love Canal area, Niagara Falls, N.Y. The purpose of the quality control procedure is to assure, assess, and document the precision, accuracy and adequacy of data developed during the program.

6.2 OPERATION CHECKS FOR ANALYTICAL METHODS

6.2.1 Calibration of Analytical Instrumentation

It is necessary that the analytical instrumentation utilized during the project be calibrated at frequent intervals, i.e., weekly. In turn, the calibration must be verified at even more frequent intervals, i.e., daily.

Calibration curves for the analyates of interest are generated weekly or more frequently as specified in the analytical protocols provided by the Prime Contractor. Calibration checks are performed every 6-8 hours of instrument operation. Mass spectrometers are tuned daily prior to analysis according to contract specifications. A summary of instrument calibration and control limits are provided in Table 6-1.

6.2.2 Analytical Method Quality Control

Quality control checks on the overall analytical methods include the use of surrogate standards, laboratory control standards (LCS), replicate analysis and method blanks to aid in the identification of systematic method and operator errors. Control charts will be maintained for each QC check where appropriate.

Specified surrogate compounds are spiked into every sample where appropriate. The surrogate compounds will be stable isotope derivatives of selected compounds that have been identified at Love Canal, as well as additional compounds that will aid in monitoring the analysis procedures.

The laboratory control standard (LCS) is a concentrate of a number of compounds of known concentration in a reference material or spiked into samples. One LCS is analyzed per sample set processed at one time, normally every 5-15 samples.

Table 6-1. Analytical Internatl Quality Control Checks

	Calibration Curve	Calibration Check	GC/MS Tuning	Surrogate Standards	Capillary Column Performance	Laboratory Control Standard	Method Blank	Replicate Analysis
Air (foam plug)	3 point, 100 fold range, each sample set	Every 6-8 hours of operation, control limit ≤±6%	Daily per method 625	Not done	Grob mixture + PCP** & benzidine tailing ≤±1.5	One analyzed per sample set, control limit ≤±2 S.D.	One analyzed per sample set	Not done
Water/soil/sediment								
Volatile organics	External standard 3 point, 100 fold range, weekly	Every 8 hours control limit ≤±20%	Daily per method 624	Added to each sample control limit ≤±2 S.D.	NA	One analyzed per sample set, control limit ≤±2 S.D.	One analyzed per sample set	One per sample set
Semivolatile organics	Internal standard response factor 3 point, 100 fold range, weekly	Every 8 hours control limit ≤±20%	Daily per method 625	Added to each sample control limit ≤±2 S.D.	Daily, grob mixture + PCP & benzidine tailing ≤±1.5	One analyzed per sample set, control limit ≤±2 S.D.	One analyzed per sample set	One per sample set
Pesticides	3 point, 100 fold range, weekly for selected set of * pesticides	Every 6-8 hours control limit ≤±6%	Daily per method 625	Not done	Confirmation only, as above	One analyzed per sample set, control limit ≤±2 S.D.	One analyzed per sample set	One per sample set

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*lindane, heptachlor, aldrin, endosulfan I, II, dieldrin, p,p'-DDT
**PCP = Pentachlorophenol
NA - Not applicable

One method blank (reagent water) is processed with each sample set analyzed. The method blank must not show any signals that correspond to compounds on the Love Canal or priority pollutants lists. If common laboratory contaminants; e.g., phthalate esters, are found in the method blanks, reported values for the same compounds in the environmental samples will be reduced by the amount found in the blank.

Intralaboratory precision of the environmental measurements is evaluated by the measurement of one duplicate (split) of a selected sample per sample set. A duplicate is a second aliquot of a sample carried through the entire method.

A summary of the quality control checks and control limits for the analytical methods is presented in Table 6-1.

6.3 CONTROL LIMITS AND CORRECTIVE ACTION

Laboratory control charts will be constructed and maintained on a number of parameters throughout the course of the analytical program (see section 6.4). The specific items of the analytical internal quality control check program with control limits and corrective actions are summarized in Table 6-2. Implementation of corrective actions in the laboratory are further described in section 8.0.

6.4 QUALITY CONTROL RECORDS MAINTAINED

Presented in this section is a list of the quality control records to be maintained and submitted along with the analytical data.

Air (foam plugs)

1. Calibration curve for quantified compounds
2* Calibration check standard data
3* Lindane peak areas
4* Recovery of internal standard
5. Peak areas for qualified compounds at single concentration
6* Recovery of laboratory control standard (LCS)

Volatile organics

Water

1. Daily tuning of GC/MS with DFTPP or BFB
2. Calibration standards for quantification
3* Daily verification of calibration
4* Surrogate compounds (2) % recovery, each sample
5* Recovery of LCS (10 compounds per sample set)
6. Comparison of replicate analysis, one per sample set

Table 6-2. Control Limits and Corrective Action

Item	Control Limit	Corrective Action
Calibration Check	$\leq \pm 6\%$ or $\leq \pm 20\%$	Generate new calibration curve. Notify task leader.
GC/MS Tuning	Multiple ion abundance ratios	Retune to meet method 624/625 criteria and repeat performance check. Notify task leader.
Surrogate Standards	$\leq \pm 2$ S.D.	Validate instrument sensitivity & calibration. Investigate to determine cause of deviation. Reanalyze samples and/or prepare justification. Notify task leader.
Method Blanks	---	If contaminants observed, notify task leader. Investigate to determine source.
Laboratory Control Standards	$\leq \pm 2$ S.D.	Validate instrument sensitivity & calibration. Investigate to determine cause. Notify task leader.
Capillary Column Performance	Tailing $\leq \pm 1.5$	Perform column maintenance procedures as directed by task leader. Repeat performance check

Volatile organics (cont'd)

Soil & Sediment

7* Additional surrogate compounds, % recovery, each sample
8* LCS for soil/sediment 99 & 95% confidence limits

Semivolatile organics

Water

1. Daily tuning of GC/MS with DFTPP or BFB
2. Calibration standards for response ratios
3* Daily verification of calibration
4. Daily analysis of grob mixture + PCP + benzidine
5* Surrogate compounds (3) % recovery, each sample
6* Recovery of laboratory control standards (15 compounds per sample set)
7. Comparison of replicate analysis, one per sample set

Soil & Sediment

8* Surrogate compounds (4) % recovery, each sample
9* LCS-NBS soil PNAs 99 & 95% confidence limits
10* Response factor variability
11.* Daily response factors on composite standard
12. Verification of internal and surrogate standard presence - each sample scan # & area

*Control chart maintained.

7.0 REPORTING MANAGEMENT SYSTEM

7.1 GENERAL INFORMATION

GSRI's internal data reporting management system is described in this section. The data origination point is the analyst who compiles the raw data package for submission to the Task Leader via the sample custodian. The function of the Task Leader is to review and verify the correctness of the data and transmit the verified data package to the document control officer. The document control officer performs the functions given earlier and assembles the final data package for submission to the GCA Technical Monitor. The Principal Investigator reviews the final data package prior to submission to GCA.

7.2 ORIGINATION OF DATA

Responsibility for acquisition and reduction of raw data resides with the analyst who performs the instrumental analysis of the sample. In addition, the analyst performs: 1) the necessary calculations to determine pollutant concentrations, 2) completes data submission forms as specified by GCA, and 3) prepares the necessary QC data pertaining to those samples he has analyzed. The sample data and QC data are then presented to the Task Leader for review.

7.3 DATA REVIEW

The function of the Task Leader in the data review system is two-fold. First, he assists the analyst in the compilation of the data package, and secondly, it is his responsibility to review the data for correctness and to verify the calculations. At this point, the data package is routed to the document control officer. The document control officer performs the functions as described in Section 4.0 and prepares the data package for submission to the Prime contractor. The final data package is then reviewed by the Project Manager who makes the actual submission to the Technical Monitor.

8.0 CORRECTIVE ACTION PROCEDURES

8.1 GENERAL INFORMATION

The purpose of this section is to describe implementation of quality control corrective action procedures in the analytical laboratory. The corrective actions are presented in Section 6.3.

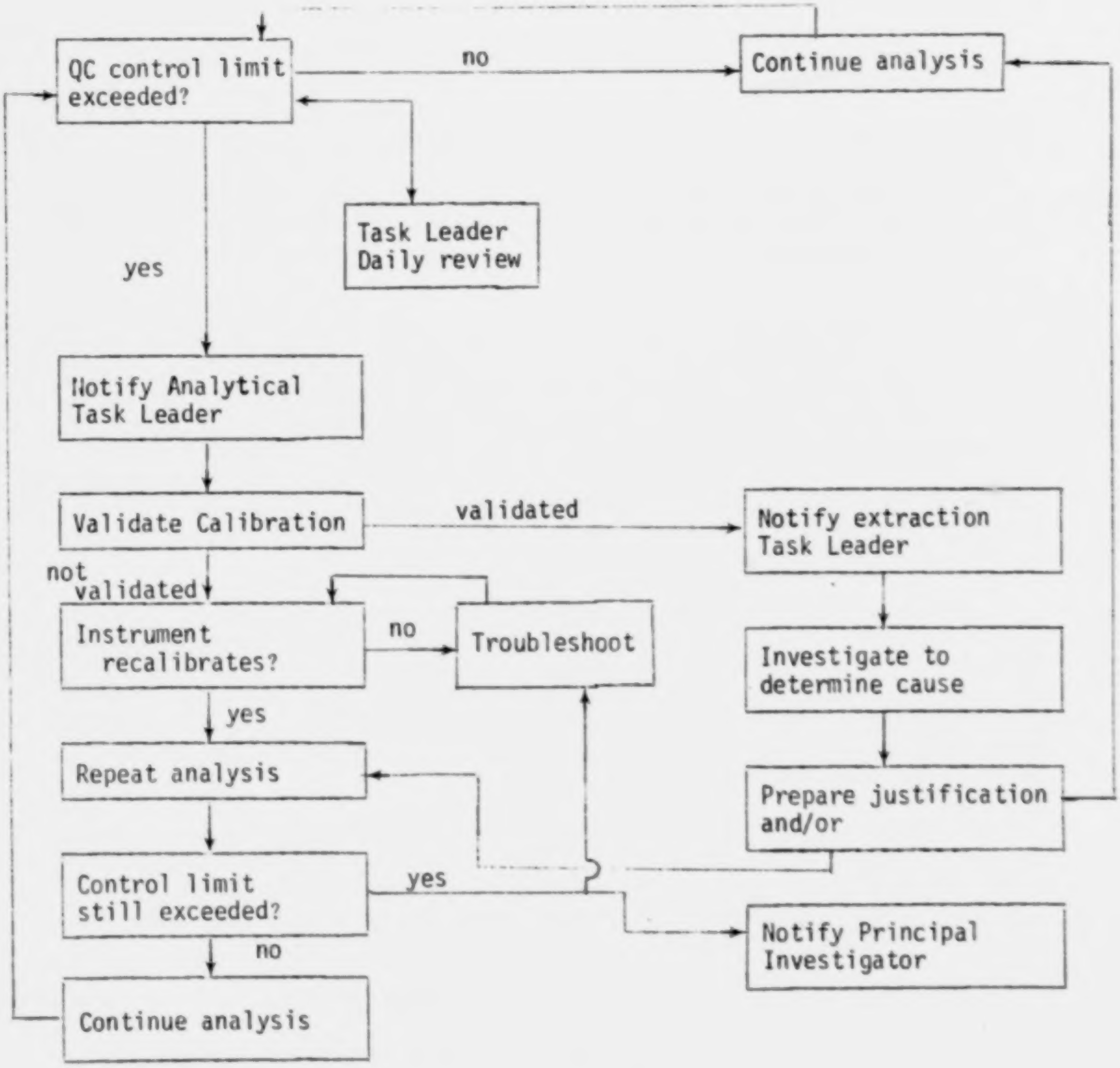
8.2 IMPLEMENTATION OF CORRECTIVE ACTIONS

Corrective actions shall be implemented according to the attached flow chart (Figure 8-1).

8.3 RESPONSIBLE PERSONNEL

The first level of responsibility for monitoring the quality control limits shall reside with the operator of the analytical instrumentation. He/She shall update the quality control charts on a per analysis basis immediately after the analysis or calibration is performed. The task leader shall also review all quality control data on a daily basis to verify that quality control limits have been met. If a quality control limit is exceeded then the operator must immediately notify the designated task leader. The task leader will then exercise the second level of responsibility which is implementation and supervision of the corrective action of choice. If, in the opinion of the task leader, the problem cannot be resolved without compromising the time schedule of the project he must immediatley notify the Principal Investigator. The Principal Investigator shall take whatever steps are necessary to ensure the continuity of the project.

Figure 8-1. Flow Chart for Corrective Actions


QC control limit exceeded?
no
Continue analysis
Task Leader Daily review
yes
Notify Analytical Task Leader
Validate Calibration
validated
Notify extraction Task Leader
not validated
Instrument recalibrates?
no
Troubleshoot
Investigate to determine cause
yes
Repeat analysis
Prepare justification and/or
Control limit still exceeded?
yes
Notify Principal Investigator
no
Continue analysis

Appendix B - Analytical Procedures

Part 2. Analytical Equipment Maintenance/Troubleshooting Procedures

1. Gas Chromatograph/Mass Spectrometer (GC/MS)

GC/MS equipment is maintained and repaired under Product Maintenance Agreement No. 4968-2 by Hewlett Packard Company service personnel. This maintenance agreement provides 48-hour response time for all service requests.

Daily measurements of performance and tuning standards determine the operating condition of GC/MS equipment. Routine troubleshooting is carried out in accordance with procedures described in the appropriate operating manuals for each instrument. In the event that routine troubleshooting procedures in the operator's manual do not bring the instrument into acceptable performance limits, operators are instructed not to proceed with sample analysis, and to inform W. L. Yauger, task leader for GC/MS analysis, that the instrument is not performing properly. Appropriate corrective action will be determined by the task leader.

For each GC/MS instrument an acquisition log is maintained in which filing reference data, date and time, run parameters, and miscellaneous data are recorded. Operating conditions, maintenance accomplished, maintenance required, tuning parameters and other pertinent data are maintained by producing hard copy from diagnostic procedures, and tuning procedures. Diagnostic programs are included in the computer software package for each instrument.

Consumable spare parts such as columns, septa, seals, pipe fittings, recording paper, syringes, samples, vials, etc. are kept on hand at all times. Spare parts involved in mechanical or electrical repairs are provided by Hewlett Packard under the terms of the maintenance contract, and are maintained in inventory at regional service centers.

2. Gas Chromatograph/Electron Capture Detector (GC/ECD)

The GC/ECD's are calibrated on a regularly scheduled basis to insure the accuracy of the data. The calibration schedule is contained in Section 6.0 of the Quality Assurance Plan. Records of calibrations, regular performance checks and service for each instrument are maintained in bound log books in such a manner that the history of performance of the instruments can be easily reviewed.

Routine maintenance is performed either according to the schedules set forth in the manufacturer's operating manual or at the analyst's discretion, whichever is most frequent. The operating manuals are kept in the laboratory in proximity to the particular instrument to which they pertain. These manuals also contain troubleshooting guides that are used by the analyst when quality control checks indicate that a problem has arisen.

Consumable spart parts are kept on hand at all times and are available from local suppliers within 24 hours. Major equipment repair items such as circuit boards, etc. are available from on-site redundant instrumentation or from the manufacturer within 48 hours.

Southwest Research Institute
QA Plan

1.0 Project Description and Organization

1.1 Project Description. This program is part of a large effort to evaluate pollutant levels present in the Love Canal area of Niagara Falls, New York. The program at Southwest Research Institute (SwRI) involves development of methodology for analysis of semivolatile organic compounds in soils and sediments, and analysis for trace organics in air, soil, and sediment samples. This work is being performed under subcontract to GCA/Technology Division, (GCA) under a prime contract with the Environmental Protection Agency.

1.2 The program will be conducted by the Department of Environmental Sciences, Division of Chemistry and Chemical Engineering. Dr. Donald E. Johnson will be the program manager and will have overall responsibility for the program. Task managers will be appointed for each of the major program elements outlined in Section 2.0.

2.0 Major Project Elements. The major project elements are as follows: development of methodology for semivolatile organics in soils and sediments; analysis of polyurethane foam plugs for pesticides and related compounds, analysis of soils and sediments for volatile and organic compounds, analysis of soils and sediments for pesticides and related compounds, analysis for benzidine in soils and sediments, qualitative GC/MS analysis, sample and document control, and quality assurance.

Methodology development will be assigned to Dr. Edward P. McGovern, who will be assisted by Mr. James M. Taylor, Mr. William A. McMahon, and Mr. Herbert J. Schattenberg, III. Msrs. Taylor, McMahon, and Schattenberg will each be responsible for the analysis by one of three candidate semivolatile methods. Analysis of soils and sediments for pesticides and related compounds will be conducted under the supervision of Dr. McGovern, with Mr. Schattenberg responsible for the

ECD determinations and Mr. Wheeler reponsible for the GC/MS quantitation. Analysis of polyurethane foam plugs for pesticides and related compounds will be under the supervision of Mr. Taylor. Analysis for benzidines will be performed by high performance liquid chromatography under Dr. McGovern's supervision, and GC/MS qualitative analysis will be performed by Mr. Wheeler. Sample control will be assigned to Mr. Taylor, who will have the responsibility for control for the keys to the sample containers and for ensuring that chain-of-custody procedures are followed. Ms. Bonnie C. Fergus will assist Mr. Taylor in the sample control, document control, and chain-of-custody aspects of the program. Mr. Richard E. Thomas will be responsible for the quality assurance aspects of the program and will assist in the document control and chain-of-custody aspects.

3.0 QA Objectives

The expected accuracy and precision to be obtained by the methodology applied is expressed in terms of percent recovery and relative standard deviation for each of the methods:

Analytical Method and Media	Precision (Relative Standard Deviation)	Accuracy (Percent Recovery)
Pesticides in Air on PUF plugs.	15	80
Pesticides in soils and sediments.	5-10	80-95
Benzidine in soil and sediments.	20	60-80
Acrolein and Acrylonitrile in soils and sediments.	20	75
Volatiles in soils and sediments.	*	†

Precision and Accuracy for semivolatiles in soil and sediments to be analyzed by the three preparative methods to be determined as the methods are evaluated.

* Precision should approach that reported by Bellar and Lichtenberg (1978).

† Accuracy should approach that reported in the "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants."

4.0 Document Control Procedures. The Document Control Officer (DCO) for the program will be Mr. James M. Taylor. Mr. Taylor will have the overall responsibility for ensuring that the procedures specified by the Prime Contractor are carried out, and that all activities conform to the procedures specified in "Enforcement Considerations for Evaluation of Uncontrolled Hazardous Waste Disposal Sites by Contractors," (EPA-330/9-78-001R).

4.1 Maintenance of Records. All records relating to the program will be maintained in a secure file, with access limited to the DCO and designed alternates. All chain-of-custody documents, notes, log books, and other documentation will be maintained in separate files, and all information entering these files will be listed in an inventory.

4.2 Personnel briefing. All personnel working on the program will be given a formal briefing prior to the initiation of work outlining their responsibilities under the program. Included in this briefing will be the requirements for custody of the samples, instructions for completion of laboratory notebooks, proper manner of making corrections to logbook entries, and instructions for secure handling of documentation. To supplement these instructions, each worker will be given a written summary of the major points, and instructed to maintain this with the analytical and QA manuals.

4.3 Internal Audits. The program manager, DCO, and/or their designated representative will perform routine, unannounced audits of the document control procedures at random intervals throughout the program. These inventories will be performed using the forms given in the policies and procedures manual. Any deficiencies will be noted and appropriate corrective action taken.

5.0 Chain-of-Custody

5.1 Responsible Personnel. The DCO will have primary responsibility for ensuring chain-of-custody procedures are followed and all documentation is properly executed. The DCO will appoint alternates to assist him and will satisfy that they understand the provisions of the procedure.

5.2 Control of Samples. Samples will be placed in a locked storage container upon receipt. The DCO and designated alternates will maintain the keys to this container and will be responsible for all samples. Analysts will sign for receipt of all samples to be processed and maintain the samples in their possession or in view at all times when the samples are outside of the storage container. When sample processing is complete, the analyst will return custody to the repsonsible personnel. At all times when custody is transferred, both the issuing and receiving parties will verify that information on the sample tag is properly recorded.

5.3 Internal Audit. At irregular intervals throughout the program the system will be subjected to an internal audit. This will consist of verifying that all samples on hand are recorded and accounted for, that custody of samples is assured in the laboratory, that all documentation has been properly executed, and that all personnel are aware of their responsibilities. Any deficiencies will be noted and corrective action taken.

6.0 Operational Checks and Correction Action

6.1 Polyurethane Foam Plugs

6.1.1 Operational Checks

6.1.1.1. Method blanks. Must not have any peaks eluting in window corresponding to any compounds on the Love Canal or priority pollutants list. One will be processed per batch.

6.1.1.2. Laboratory Control Standards will be within established limit set by five replicate analyses of blank plugs spiked with LCS compounds at the 250-400 ng/whole plug level. [2,4,5-Trichlorophenol will be analyzed by HPLC.] One LCS will be processed per batch.

6.1.1.3. Lindane must provide 50% full-scale deflection at the 225 ng injected level. [With EC detector.]

6.1.1.4. 50 pg of each component must be quantifiable by GC/EC and HPLC.

6.1.1.5. A three point calibration curve containing an internal standard (dinitro benzene, dibromobenzene, 2,2'-dichloro-p-xylene are possibilities) will be prepared daily. Calibration will be verified every 5th sample and must be within ± 5% of the actual value.

6.1.2 Corrective Action

6.1.2.1. Subtract blank value from sample value. Make certain glassware is clean. Repeat to check for possible intralaboratory contamination. Check solvents.

6.1.2.2. Check soxhlet extraction set up to make sure sample goes theough at least 50 solvent cycles. Check cooling water, heaters, etc. Check volume of final extract. Determine if procedural error occurred during concentration step.

6.1.2.3. Check E.C. overall condition. Clean detector if dirty. Check carrier gases, examine septum for leaks. For HPLC, check solvent purity, condition of electrode, repolish and recondition if necessary. Check for solvent leaks. Check cell voltage and readjust if necessary.

6.2 Base Neutrals and Acids

6.2.1 Operational Checks and Corrective Action

6.2.1.1. Tune M. S. using DFTPP. The instrument must meet the specifications given in Method 625, Federal Register, Monday, December 3, 1979, page 69546. If criteria are not met adjust instrument according to manufacturers procedure.

6.2.1.2. Response factors for semivolatiles and pesticides are determined following Method 625, Section 6.3, page 69541, and must be verified daily. If drift occurs an entirely new calibration curve must be constructed and samples reanalyzed, (i.e, when

Rf values for naphthalene, anthracene and chrysene differ from the initially determined mean values by ± 1.5 times, new Rf values at 3 levels will be determined).

6.2.2.3. Performance of 30M SE54 00.25-mm ID. fused quartz capillary column must be verified by daily injection of 100 ng of benzidine and pentachlorophenol simultaneously in a 1.0 μL injection. The tailing factor must be ≤ 1.5. Corrective action: clean injector, check column temperature, transfer lines. Replace capillary column.

6.2.2.4. Process one method blank per sample set extracted. If contamination other than phthalates appears, reprocess samples using refired glassware, new solvents and freshly prepared alumina, florisil, etc.

6.2.2.5. Laboratory control charts will be prepared by initially spiking 4 replicate 5g portions of NBS SRM-1645 river sediment with the Acid and B/N LCS standards. Once the means and standard deviations have been determined, one LCS will be processed every sample batch. If a value falls outside the control limits, the cause will be determined, corrected and samples reanalyzed.

6.2.2.6. Surrogate compound data will provide information about the variation of extraction efficiency between samples.

6.2.2.7. Every tenth sample will be split and analyzed in duplicate.

6.3 Soils and Sediments, Base Neutrals - Benzidine

6.3.1. Operational Checks and Corrective Action

6.3.1.1. If $(GC)^2$/MS indicates the presence of benzidine, it will be quantified using Method 605 employing HPLC/electrochemical detection.

6.3.1.2. Method blanks will be analyzed. One method blank will be processed per batch.

6.3.1.3. Control charts using benzidine spiked onto the NBS river sediment and carried through the sample homogenization/extraction will be constructed by analyzing 4 replicate samples. One LCS per batch will be analyzed after this initial set up.

6.3.1.4. Standards will be analyzed over two orders of magnitude and a single concentration standard reanalyzed every 5 samples.

6.3.1.5. The performance of the HPLC/Electrochemical detector combination will be checked daily by injection of 3 standard benzidine concentrations. The cell voltage will be checked daily and adjusted as necessary.

6.4 Soils and Sediments, Volatile Organics

6.4.1 Operational Checks and Corrective Action

6.4.1.1. Three replicate analyses on 1g aliquots of NBS 1645 soil spiked at the 100 ppb level with benzene, toluene and chlorobenzene will be performed to establish control charts. One LCS will then be analyzed every batch. Surrogates will also be spiked into these samples.

6.4.1.2. Every 10 samples a duplicate analysis will be performed on one sample.

6.4.1.3. One method blank using organic free water will be analyzed every 10 samples and must not show any contamination with compounds on the Love Canal list or priority pollutant.

6.4.1.4. The internal standards methyl chloride-D_2-, benzene-D_6, and toluene-D_8 in organic free water will be added to the water diluted samples.

6.4.1.5. The surrogates will be spiked directly onto the soil or sediment in the 40-mL purging vial before the addition of the water.

6.4.1.6. If analysis by Method 624 indicates presence of acrolein or acrylonitrile, a second sample will be analyzed using Method 603. This will require spiking an additional sample to determine recoveries from the sample. Purging efficiencies and analytical precision will be determined according to the procedure set forth in Method 603, Section 6.

6.4.1.7. The GC/MS system will be tuned daily as set forth in Method 624 using DFTPP. The performance criteria must be met before response factors are determined or any samples are processed.

6.4.1.8. The internal standards and surrogates will be analyzed to establish response factors. Response factors will then be determined for all compounds on the Love Canal list using the supplied Supelco purgeable standards. The response factors for the surrogates will be determined daily.

6.5 Soils and Sediments, Pesticides

6.5.1. Operational Checks and Corrective Action

6.5.1.1. One method blank will be processed per batch to insure glassware is contamination free.

6.5.1.2. Every tenth sample will be analyzed in duplicate.

6.5.1.3. Calibration standards containing all pesticides of interest will be prepared, appropriate dilutions made and the instrument linearity range defined (as per Method 608, Federal Register, page 69502). The exception to this will be the Aroclors which will be analyzed separately.

6.5.1.4. A single mixed standard will be analyzed every batch to check for instrument variation. If a problem is indicated, detector will be examined for contamination and entire system checked for leaks, purity of carrier gases, human error or injection volume.

6.5.1.5. Laboratory control charts will be constructed for aldrin, dieldrin, and heptachlor by initially spiking 3 replicate SRM 1645 sediment samples at the 5 ppm level and processing through the entire method. One LCS will be processed with each batch analyzed.

6.5.1.6. Surrogates will be added to each sample, including LCS, at the 5 ppm level. (Tetrachlorobenzene $^{13}C_6$, Hecachlorobenzene $^{13}C_6$; p-chloro-toluene-d_6 unless otherwise directed).

6.5.1.7. Every fifth sample will be spiked with Method 608 organochlorine pesticides and one Aroclor for the determination of recoveries.

7.0 Data Reporting Management System

7.1 Data Recording Forms. The data forms provided by the Prime Contractor will be used for the recording of all data generated on the program. Each analyst will be provided with the analytical data sheets pertinent to the analysis being performed, and will execute these upon completion of the analysis. The analyst will be responsible for reviewing the data as it is generated, to ensure that the results are reasonable and interpretable, and that all information required for recalculation of the results is available. The analytical data sheets, chromatograms or spectra, and supporting information will be supplied to the responsible individual for review.

7.2 Data Review. The individual responsible for each analytical subtask will perform a review of all data before execition of the data encoding forms. This review will consist of the following elements: review of analytical program, verification of GC data, examination of results, verification of results.

7.2.1. Review of Analytical Program. The responsible individual will review each set of results to ensure that the required program elements, including method blanks, surrogate spikes, and QC/SRM samples, have been accomplished according to the program design. The supporting data will be reviewed to ensure that analyses were performed under the correct conditions and that all required procedural steps were performed.

7.2.2. Verification of QC Data. Quality control data will be reviewed to ensure that the laboratory control samples were within the control limits and that method blanks satisfied the acceptability criteria.

7.2.3. Examination of Results. The reponsibile individual will examine the results obtained along with the pertinent chromatograms or spectra to ensure that the results are reasonable and that the analyst has interpreted the results correctly. Any unusual or unexpected results will be reviewed and a resolution will be made as to whether or not the analysis should be repeated or the result recalculated.

7.2.4. Reculaculation of Results. The responsible individual will selectively recalculate results to ensure that the calculation was performed properly. Emphasis will be placed on those results showing a significant concentration of one or more of the analytes of interest. As a minimum, one set of calculations will be checked for each lot of samples analyzed.

7.3. Data Encoding Forms. Following the data review, the verified data will be transferred from the analytical data sheets to the data encoding forms. The Document Control Officer will be responsible for ensuring that all required information is recorded and for reviewing the completed form for the accuracy of the transferred information. All data will be verified against the analytical data sheets, and the completed forms will be reviewed by the individual responsible for the analysis prior to submission of the results to the Prime Contractor.

8.0 Corrective Action Procedures. If problems arise, Dr. Edward P. McGovern will immediately be informed. He has the responsibility for maintaining all gas chromatographic, HPLC and mass spectrometry equipment. This will entail daily monitoring of all standards analyzed on all instrumentation to ascertain if the instrumentation is functioning properly and if not initiate corrective action.

Mr. Herbert G. Wheeler will be the primary mass spectrometer operator and will perform the instrument tuning and calibraiton, and actual sample analysis. He will be in constant communiction with Dr. McGovern.

APPENDIX B

Part 2. Analytical Equipment Maintenance Troubleshooting Procedures

1). GC/MS

In-house mainenance and troubleshooting will be performed by Mr. Herbert G. Wheeler, the mass spectrometer operator and mass spectrometry lab supervisor.

Finnigan Instrument Corporation has provided its customers with maintenance and troubleshooting guids and if we cannot correct a problem in-house we have a current maintenance contract with Finnigan.

2). GC-EC/HPLC

In-house maintenance of the GC-EC will be performed by Dr. Edward P. McGovern following the manufacturers (Tracor, Hewlett Packard) recommendation provided with each instrument.

HPLC maintenance/trouble shooting will follow Waters recommended schedules and procedures. The electrochemical cell will be checked daily and if necessary reconditioned following procedures recommended by Bioanalytical Systems, Inc.

3). Analytical Balances

Analytical Service Company in Dallas, Texas provides yearly calibration and service for our analytical balances. Daily balance stability is monitored by determining the weight of an internal laboratory standard mass and plotting the results on a control chart.

4). Spare parts inventories of parts subject to normal wear replacement are maintained for GC/MS, GC/EC; HPLC.

GC/MS: filament
ion multispheres
spare source
spare vacuum pumps
spare set of rods

GC/EC: Spare detectors
fittings
valves
flow controllers

Due primarily to the fact we have seven EC equipped instruments.

HPLC: Spare columns
fittings
pumps
electrode reconditioning kits

Compuchem/Mead Technology Laboratories

Revised QA Plan

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INTRODUCTION

In response to GCA's request, COMPU/CHEM has prepared a revised Quality Assurance Plan which more closely follows the format referenced in the subcontract document.

This presentation of COMPU/CHEM procedures places emphasis on generating defensible analytical data. To achieve this objective, three performance criteria are introduced into the program The three elements are:

1) Chain of Custody Documentation
2) Scheduling and Control
3) Data Validation via Quality Control Practices

The significance of each item as well as the performance criteria are detailed as part of this presentation.

1.0 PROJECT DESCRIPTION AND ORGANIZATION

The purpose of this program is to act in support of our client, GCA Corporation, in the performance of their prime contract. As a subcontractor, COMPU/CHEM will supply analytical data obtained from environmental samples analyzed during the period August 20, 1980 thru November 30, 1980. These samples (soil, sediment, water) will be analyzed for trace levels of organic compounds.

As emphasized in the original solicitation, the successful accomplishment of this program will be measured by the degree to which subcontracting laboratories can document sample integrity and authenticity of data. These considerations were re-emphasized during meetings held in Las Vegas, August 19, 1980.

In response to these objectives, COMPU/CHEM has committed sufficient human resources and capital equipment to accomplish all required tasks within the established period of performance. Table #1 identifies personnel who play a key role in the performance of this contract.

TABLE #1

Personnel	Program Responsibilities
K. McConnaghy	Program Manager
R. L. Myers	Vice President
J. Russell	Director of Technical Affairs
J. Zoldak	GC/MS Laboratory Manager
P. Ragsdale	Quality Control Manager
M. Cartegena	Chain-of-Custody Custodian
R. Meierer	Scheduling and Control

2.0 MAJOR PROGRAM ELEMENTS

This quality assurance program requires the simultaneous performance of three (3) major elements.

1) Chain-of-Custody Documentation
2) Scheduling and Control
3) Data Validation via Quality Control Practices

The following is a description of COMPU/CHEM's approach to ensure the accomplishment of these three elements.

1. M. Cartegena has responsibility for the coordination of chain-of-custody documentation. M. Cartegena will introduce each GCA sample into the COMPU/CHEM system. This begins with physical inspection of each sample upon receipt. Physical inspection is performed in conjunction with documentation correlation (i.e., sample container number matches chain-of-custody number and description of container). Verification is indicated by signing the chain-of-custody document. GCA samples are then assigned a COMPU/CHEM number. This four digit number is unique within the COMPU/CHEM system. Once this number is assigned, a folder is prepared for each sample. The chain-of-custody record is placed in the folder as well as all information pertaining to that specific sample. Samples containers are labelled and stored in a secured refrigerated area. Logs are kept in each working area for the purpose of identifying the person, date, and time during which sample is not in the secured refrigerator. Chain-of-custody procedures, Section 5, present greater detail of the specific procedures.

2. Once a sample has been given a COMPU/CHEM number, the Scheduling and Control office prepares a coded sequence of instructions from which work lists are prepared. These work lists provide the technical staff

with detailed operational parameters including specific instrumentation and analytical protocols. The control system augments the chain-of-custody logs by identifying work stations at which tasks are performed. Strict adherance to scheduling allows COMPU/CHEM to control samples by performance and by exception. This practice facilitates early identification unanticipated performance issues. This element of our program is further detailed in Sections 4.0, 7.0, and 8.0

3. The introduction of Quality Control via standards, surrogates, blanks and duplicates is primary to data validation. Due to the special emphasis placed on QC procedures, P. Ragsdale will introduce, monitor and report all QC practices. A detailed presentation of these requirements can be found in Section 6.0.

3.0 QA OBJECTIVES

The objectives of this program are:

- provide GCA with sufficient QC data to support the quality of the analytical data delivered.
- document sample integrity.

The objectives identified above have been expanded upon in other sections of this plan. Briefly, Table #2, identifies the precision and accuracy parameters to be achieved during the course of this program.

Table #2

Method	Media	Accuracy	Precision
Volatiles	Water	90%	± 10%
Volatiles	Soil/Sediment	80%	± 20%
Acid Extractables	Water	50%	± 10%
Acid Extractables	Soil/Sediment	40%	± 15%
B/N Extractables	Water	80%	± 10%
B/N Extractables	Soil/Sediment	70%	± 15%

4.0 DOCUMENT CONTROL PROCEDURES

In the performance of this contract, the Program Manager will act as the focal point for document control. Documents to be managed included:

1) Subcontract #1-619-026-222-002E
2) Modifications to the subcontract
3) Amendments, revisions, clarifications to the subcontract
4) Original Solicitation
5) Chain-of-Custody records
6) Telephone communications log
7) Laboratory logs
8) Shipping and receiving records
9) Reporting instruments supplied by GCA
10) Laboratory results

Items 1, 2, 3, and 4 are maintained by the Program Manager and by COMPU/CHEM's contract specialists. Items 5 and 9 are under the supervision of the Chain-of-Custody Custodian. Item 6 will be the sole responsibility of the Program Manager. Items 7 and 8 are not unique to the GCA program and are available for inspection only. Item 10 is monitored by the Scheduling and Control department.

5.0 CHAIN-OF-CUSTODY

GCA samples are of an evidentiary nature and must be traceable from the time a sample is collected until analysis is complete. In addition, samples must be secured at all times.

For this program, the following procedures have been established:

1) Receipt of sample is authenicated by the Sample Custodian's signature.
2) Samples are stored in a locked refrigerator until analytical phase begin. (A log is maintained for entry and exit.)
3) All entrances to COMPU/CHEM are secured.
4) Visitors to COMPU/CHEM are escorted while on the premises.
5) Samples are either in a locked refrigerated area or in the possession of an analyst, in which case, the laboratory log identifies the person, sample number, and time.
6) Chain-of-custody records are maintained in individual sample folders and returned with the report of data.

6.0 LABORATORY DATA CONTROL

Extractions

At the time of receipt, all information (sample number, matrix, test number required, and sample conditions) required by the extractions laboratory for a sample is entered in the extractions log. All water extractions are scheduled to begin within 48 hours of receipt. Other matrices may require additional preparation and are not necessarily subject to the time limit. The individual methods contain the analysis requirements for the various matrices. At the time of extraction, the date and volume or weight of the sample extracted are recorded in the log under the appropriate test number column. The code number and volume of any surrogate or spiking standards added to the sample are recorded also with the extractor's initials. As a sample is extracted, all glassware and each extract is labelled with the sample number and appropriate test number. Both codes are required to insure each extract receives the proper analysis.

An extractions notebook is maintained for recording daily operating conditions. The reagent lot numbers used each day are recorded with the associated sample numbers. Any problems encountered in the extraction of a sample are recorded in the notebook, as well as data associated with any samples receiving special preparation.

Instrumentation

Each instrument is assigned a code number for identity. The code numbers are unique to each instrument, permanent, and used for all instrument logs and records. Each instrument has a maintenance log assigned to it. Column and septa changes are recorded in the logs, as well as other maintenance records.

a. GC/MS: run logs for the mass spectromenters are maintained by analytical test numbers, with the date, sample number, test number, analyst, instrument number and data disk number listed for each entry. A separate page of the run log is used for each instrument and each shift. Instrument parameters are checked at the beginning of each shift, and a copy of these parameters are maintained in a separate file. All chromatograms are labelled with the sample number, test number, instrument code, the volume injected, and the analyst's name or initials. The GC/MS data system provides the data and time of analysis. Standards and calibration runs are identified by the following format:

ID / Date / Shift Code / Instrument Number

where:

ID = ACSTD, BNSTD, VOASTD, DFTPP, or PFBB

Date = a 3-digit month and day code

Shift Code = A for 1st Shift; B for 2nd Shift: C for 3rd Shift.

All chromatograms (sample and standard), and calibrations are forwarded to the Quality Control Manager for review, and the Data Management for filing.

b. Gas Chromatographs: each gas chromatograph has an assigned run log/ notebook. These notebooks are used to record the sample order in the autosampler racks, column and daily operations information, and sample calculations. Chromatograms are labelled with the date and run log number/page number. A separate work sheet is generated at the time of extraction for pesticide samples. This form is completed with volume of sample extracted, and extract volume in the Extractions Laboratory. During the pesticide analysis, all dates analyzed (with log page references), all clean-up measures performed, and all analytical data, are entered on the work sheet. The completed pesticide worksheet is forwarded to Data Management for storage in the sample files.

Standards

All standards are prepared with the utmost care by experienced laboratory personnel. A five-place balance mounted on a marble table is used in the preparation of all standards, except those involving extremely hazardous compounds which require the four-place balance located inside the glove box. All standards prepared for use throughout the laboratory are assigned a four-digit code number. The standard code number is entered in the bound standards notebook with all information regarding the preparation of that standard--i.e., date, analyst, name of each compound and amount used, final volume, and solvent used. All standards containers are labelled with the standard's code, date and analyst's initials.

Base-Neutral Standards

Base-Neutral stock standards (1.0 mg/ml of each compound) are prepared every six weeks, except stock standards of benzidine, n-nitrosodi-n-propylamine and 3,3'-dichlorobenzidine, which are prepared every two weeks. The working base-neutral standards are prepared from the stock solutions at the beginning of each week. All base-neutral standard preparations are stored in amber, ray-sorb, or foil covered flask containers and are kept refrigerated when not in use.

Acid Standards

Acid stock standards (1.9 mg.ml of each compound) are prepared every six weeks. The working standards are prepared from the stock solutions each week. All acid standard preparations are stored in glass containers and are kept refrigerated when not in use.

Volatile Standards

The volatile stock standards (0.2 mg/ml of each compound) are purchased sealed in glass ampules. New ampules are opened each week for use in preparing working standards. The working standards are prepared twice each week. A volatile surrogate stock standard is prepared in-house every four weeks. The working solution of this standard is prepared twice each week. All working strength, internal, and surrogate standards are stored in vials sealed with mininert caps. All volatile standards are kept refrigerated or in an icebath at all times.

Pesticide Standards

Pesticide stock standards are prepared quarterly. The working standards are prepared every four weeks. All pesticide standard preparations are stored refrigerated in glass vials sealed with teflon lined crimp caps.

d_{10}-Anthracene Standard

A 5 ug/ul d_{10}-anthracene standard--the internal standard for base-neutral and acid analysis--is prepared at the beginning of each week by weighing 0.05000 g ± 0.00010 and diluting to volume in a 10 ml volumetric flask. Prior to use each d_{10}-anthracene standard is analyzed on one or more GC/MS systems. The response obtained per 100 ng is compared with the response obtained for the previous standard on the same instrument that day. The two standards must agree within 10%. After the data have been reviewed by the Quality Control Officer, and all requirements met, the new standard is released for use. Each released standard bears an expiration date. After that date, the standard is not discarded, but is may not be used.

Acid Surrogate Standard

An acid surrogate standard is prepared every two weeks for use with samples. The routine acid surrogate solution is prepared by weighing 0.01000 g ± 0.00010 each of d_6-phenol and 2-fluorophenol, and quantatively transferring both compounds to a 10 ml volumetric flask for dilution to volume. Prior to use by the Extractions Laboratory, each acid surrogate standard is analyzed on one of more GC/MS systems. The response obtained per 200 ng is compared with the response obtained for the previous standard on the same instrument that day. The two standards must agree within 10 percent. After the data has been revised by the Quality Control Manager, and all requirements met, the new standard is released to the Extractions Laboratory for use. Each released standard bears an expiration date. After that date, the standard is not discarded, but it may not be used by the Extractions Laboratory.

Base-Neutral Surrogate Standard

A base-neutral surrogate standard is prepared every two weeks for use with samples. The routine base-neutral surrogate solution is prepared by weighing 0.01000 g ± 0.00010 each of d_5-nitrobenzene and 2-fluorobiphenyl and quantitatively transferring both compounds to a 10 ml volumetric flask for dilution to volume. Prior to use by the Extraction Laboratory, each base-neutral surrogate standard is analyzed on one or more GC/MS systems. The response obtained per 200 ng is compared with the response obtained for the previous standard on the same instrument that day. The two standards must agree with 10 percent. After the data have been reviewed by the Quality Control Manager, and all requirements met, the new standard is released to the Extractions Laboratory for use. Each released standard bears an expiration date. After that date, the standard is not discarded, but it may not be used by the Extractions Laboratory.

7.0 DATA REPORTING MANAGEMENT SYSTEM

Reports of data for this program will be preceeded by a three (3) step procedure prior release. The steps include:

1) Review of QC data by the QC Manager
2) Review of sample data by the Laboratory Manager
3) Review of QC and Sample Data by a Technical Expert

Following the review, the analytical data is formatted and reviewed by the Program Manager for completion in terms of contract deliverables.

8.0 CORRECTIVE ACTION

In the event of chain-of-custody errors, laboratory accidents, protocol interpretations, operation, difficulties or QC guidelines, COMPU/CHEM will follow the following chain of events to determine the appropriate corrective action:

1) Reference contract documents and supplemental information received from GCA.
2) Contact appropriate party at GCA.
    - Ken McGregor, Technical & Administration matters
    - Mary Atkinson, Contract matters
    - Mary Havelock, Report formatting
    - Rosemary Ellersick, QC
    - Stephanie Diamond, Chain-of-Custody
    - Paul Mills*, QC Materials

*EPA, EMSL-LV by referral only except in this case.

All program performance issues will be addressed as part of our routine progress reports which accompany reports of data.

PJB/Jacobs Engineering Group, Inc.
QA Plan

JACOBS ENGINEERING GROUP INC.

251 SOUTH LAKE AVENUE PASADENA, CALIFORNIA 91101
TELEPHONE (213) 449-2171 (213) 681-3781 TELEX 67-5458

Quality Assurance Plans for Analysis of Samples from Love Canal

1.0 Project Description

The project involves analyses of water, soil, and sediment samples for organic and inorganic species listed in appendix A, according to the methods specified by the prime contractor. Several departments within the laboratory will be involved in the performance of the analyses and in the maintenance of samples, extracted fractions, records, and data. These departments are:

a. Sample control center
b. Extraction Laboratory
c. GC/MS and GC Laboratory
d. Instrument Laboratory
e. General Laboratory

The department heads will report directly to the Project Manager, who will be responsible for all communications with the prime contractor.

2.0 Project Elements

The project is conveniently divided into the following elements.

a. Sample receipt and document initiation
b. Sample preparation
    i) Extractions for organics
    ii) Digestion or leaching for inorganics
c. Sample analysis
    i) GC and GC/MS for organics
    ii) AA for metals
    iii) Fluride and Nitrate
d. Data Reduction
e. Review of Surrogate Recovery or Spike Recovery data
f. Corrective action if necessary
g. Data reporting
h. Storage or shipment of residual samples and fractions.

Several of these elements will be further discussed in Sections 3.0 and 4.0

3.0 Document and Sample Control, Data Reporting

3.1 Sample Receipt

Samples will be received by the Sample Control Center accompanied by the Chain-of-Custody documents. The Sample Control Center will count the number of samples, compare it with the chain-of-custody records, note any exceptions, and sign the chain-of-custody documents. Should any exceptions be noted (i.e., fewer samples than shown on documents, more samples than shown on documents, broken samples, spilled samples), the Sample Control Officer will immediately notify the Program Manager, who will in turn notify the Prime Contractor.

The Sample Control Officer will prepare an internal traveller to accompany the samples. The traveller will show the following information: a Laboratory number, the name of the client, the number of samples, type of sample (water, soil, sediment), the identification on the sample, and the required analyses. Each sample will then be tagged using the traveller number followed serially by the sample number. The samples are then transferred to a secured refrigerator. Copies of the traveller are distributed to the individual laboratories.

When the samples are to be prepared for analysis, the manager of the extraction laboratory or a person designated by him will request the samples from the Sample Control Officer. The Sample Control Officer will issue the samples and note on the traveller the date of sample transfer to sample preparation. When the samples are prepared, the extracts and/or digests as well as the excess unused samples (or empty containers) will be returned to the Sample Control Officer. The Sample Control Officer will verify that all samples and extracts are accounted for, and note the date of completion on the traveller. He will then transfer the extracts or digests to the appropriate laboratory and the remaining samples into the secured sample storage.

3.2 Log Books

Each laboratory involved in the processing and analysis of the samples will maintain a separate log book to record the necessary information. The entries will be in chronological order. Every page of the log book must be numbered, dated, and signed. All entries must be in ink. There must be no erasures; if a mistake in entry is made, the error is crossed out and initialed.

The minimum information that must be recorded is itemized below:

3.2.1. Extraction Log Book

a) Traveller number
b) Sample number
c) Sample type (water, soil, sediment)
d) Method of extraction
e) Sample Size
f) List of Spikes and Surrogates
g) Concentration of Spikes and Surrogates
h) Final Volume of extract

3.2.2. GC/MS Log Book

a) Traveller number
b) Sample number
c) Tuning data file number
d) Calibration data file number
e) Type of extract (base/neutral, acid, combined)
f) Fraction data file number
g) Volume of extract injected
h) Chromatographic conditions

3.2.3 GC Log Book

a) Traveller number
b) Sample number
c) GC run number
d) Calibration run number
e) Volume injected
f) Chromatographic conditions (column, detector, temperature programming)

3.2.4 Metals Analysis Log Book

a) Traveller number
b) Sample number
c) Type of sample
d) Method of sample preparation
e) Sample size
f) Final volume of prepared sample
g) AA run numbers for each metal
h) AA run numbers for each metal calibration
i) List of spikes and spike concentration

4.1.2 GC

The gas chromatographic analysis for pesticides will follow the QC requirements of Method 608 for calibration and standardization.

For each set of samples prepared and analyzed at the same time a method blank will be run, as well as a spiked method blank. The frequency of replicate runs will be one sample per ten samples. If a given batch of samples is smaller than ten, a single sample of the batch will be run in duplicate.

2.1.3 Atomic Absorption

The following table summarizes our quality control steps followed in metal analysis.

Procedure	Frequency	Comments
Method blank	Once for every set of samples prepared and analyzed at the same time.	------
Calibration Standards	Three standards for each metal at the beginning of a set of samples, and after every tenth sample	Establish linear response range
Spikes	First 10 samples of each type of water; followed by one sample in a set of 20 samples	Recoveries must correspond to 85-115% of the true spike level, otherwise use method of standard additions
Duplicate runs	One in every 10 samples	---------
Laboratory Control Standards	Once for every set of samples prepared and analyzed at the same time. At least once per day	Prepare and maintain control charts

3.2.5 Fluoride and Nitrate Log

a) Traveller number
b) Sample number
c) Sample size
d) Calibration data

3.3 Data Reporting Forms

In each department the raw data will be converted to concentrations in the sample and summarized on data reporting forms. The results accompanied by the hard copies of the raw data will be transmitted for review to the department manager or another senior scientist designated by the department manager. This person will review the results and check the quality control data, such as spike recoveries, against the control charts. The data reporting forms with the extracts and/or digests will be returned to Sample Control Officer.

The sample Control Officer will verify that all required analyses have been reported. He will also confirm that all extracts and/or digests have been returned. The Sample Control Officer will store the prepared fractions and transfer the data reporting forms to the Program Manager.

3.4 Data Sheets

The program manager or a person designated by him will transfer the data from the data reporting forms to the data sheets and transmit the data sheets to the prime contractor.

4.0 Analytical Quality Control Procedures

4.1 Instrumental and Method Calibration

4.1.1 GC/MS

The following table summarizes the procedures

Procedure	Compound	Frequency	Comments
a) Tuning of Mass spectrometer	DFTPP	Daily	Must meet criteria set in Method 625
b) GC/MS Performance	Benzidine and/or pentachlorophenol	Daily	Must meet criteria set in Method 625
c) Initial Calibration of GC/MS	Standard mixture of listed components	Beginning of program	Establish working concentration range (3 point) and response factors
d) Routine calibration of GC/MS	Standard mixture of listed components	Every 8 hours	Determine response factors. Allow no more than 20% deviation
e) Method Blank	None	One for every set of samples prepared and run at the same time (max. 10 samples)	All components of interest must be below 10 ppb
f) Spiked blanks	Laboratory Control Standards	For every set of samples prepared at the same time	Recovery must be in the range-50% to + 100% maintain control charts
g) Surrogates	Supplied by prime contractor	Every sample	Recovery must be in the range-50% to + 100%. Maintain control charts
h) Replicates		One sample in every ten	Sample split in laboratory and entire analysis done in duplicate
i) Internal Standards	d_8-Naphthalene d_{10}-Authracene d_{12}-Chrysene	Every fraction of semivolalile components	

4.1.4 Fluoride and Nitrate analysis

The following table summarizes the quality control requirements

Procedure	Frequency	Comments
Method blank	Once for every set of samples prepared and analyzed at the same time	-----
Calibration Standards	Three standards at the beginning of a set of samples, and after every tenth sample	Establish linear response range
Spikes	First 10 samples of each type of water; followed by one sample in a set of 20 samples	Recoveries must correspond to 85-115% of true value; otherwise use method of standard additions
Duplicate Runs	One in every 10 samples	-----
Laboratory Control Standards	Once for every set of samples prepared and analyzed at the same time. At least once per day.	Prepare and maintain control charts.

4.2 Corrective Actions

Should the instruments or methods appear to be out of control, as measured by the laboratory control standards, duplicate runs, and calibration runs, the department manager must be alerted. After correcting the situation, the previously analyzed samples will be reanalyzed in reverse order, without repreparing the samples. If reanalysis indicates that the originally obtained results are correct, the sample preparation step will be repeated, and the newly prepared samples analyzed.

If a delay in reporting results is anticipated due to repeated analyses, the program manager will be alerted, so that he can inform the prime contractor of the delay.

Appendix Q
Plan 4.7

TRW, Inc.
QA Plan

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1.0 PROJECT DESCRIPTION AND ORGANIZATION

Under subcontract #1-619-026-222-010 TRW is analyzing 50 water samples for Love Canal priority pollutants using the techniques: electron capture gas chromatography for pesticide determination, volatile organic analysis by purge and trap GC/MS and semivolatile organic analysis by fused silica capillary GC/MS. The organization of the overall project is presented in Figure 1-1. Responsible individuals for each of the major elements are presented in the Project Organization Chart.

The analyses are being conducted within a very limited time frame and the data transmitted to GCA on previously submitted reporting sheets. The quality assurance implemented by TRW as a part of the subcontract is based on the requirements in the subcontract which in turn is based primarily on EPA requirements as a part of Federal Register Methods 608, 624 and 625. An abbreviated description of these various QA/QC requirements and implementation procedures are included in this plan.

2.0 MAJOR PROJECT ELEMENTS

Each of the individual major project elements are presented in Figure 1-1 together with the individuals responsible for performance of that particular element. A brief explanation of each element is given below:

- The sample preparation area is responsible for the extractive preparation of samples for subsequent semivolatile organic analysis and for pesticide analysis using Methods 625 and 608 respectively.
- The sample control center personnel log and distribute the samples and monitor their movement through the laboratory.
- The volatile organic analysis area is responsible for GC/MS analysis of samples by purge and trap.
- The semivolatile organic analysis element of the organization is responsible for fused silica capillary GC/MS on combined fractions.
- The pesticide analysis area uses electron capture gas chromatography for analysis with confirmation by GC/MS where possible.
- The document control office monitors all aspects of documentation including reporting.

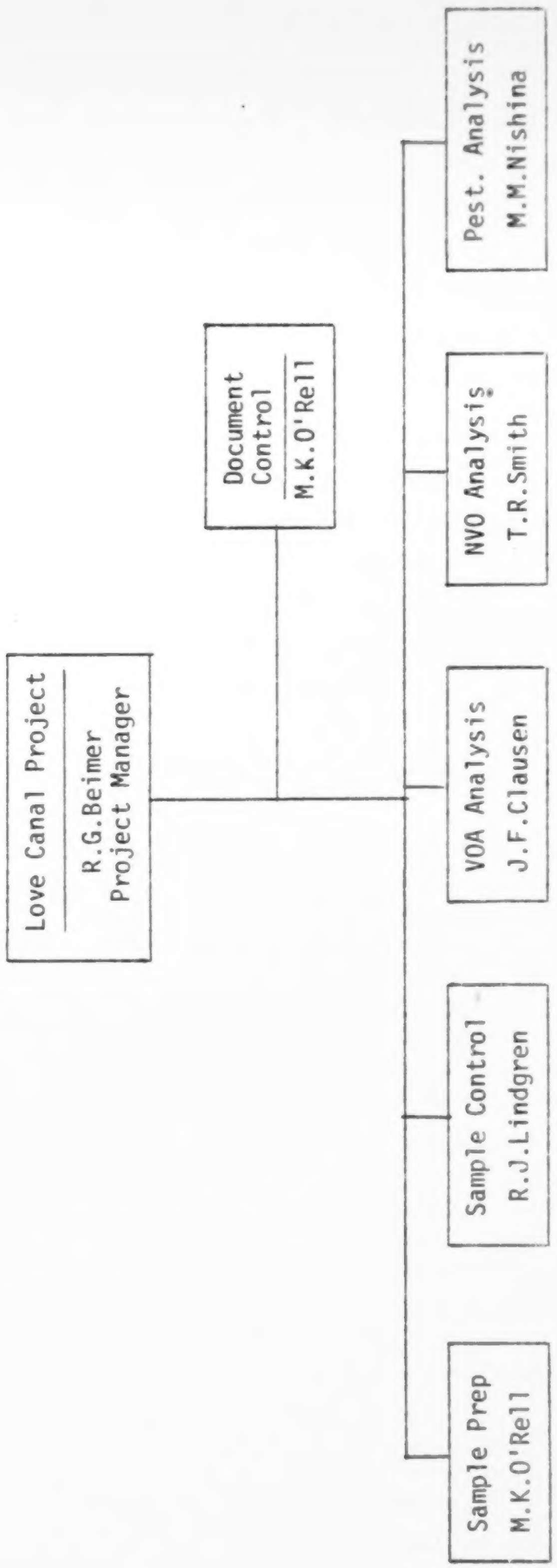
Love Canal Project
R.G.Beimer
Project Manager
Document Control
M.K.O'Rell
Sample Prep
M.K.O'Rell
Sample Control
R.J.Lindgren
VOA Analysis
J.F.Clausen
NVO Analysis*
T.R.Smith
Pest. Analysis
M.M.Nishina

Figure 1-1. Love Canal Project Organization.

3.0 QA OBJECTIVES

The quality assurance objectives of the laboratory are specific to each of the analytical methods employed. The objectives for precision and accuracy are monitored by a means of the laboratory control standard samples and duplicates together with various instrument calibration monitoring techniques which are described in detail in Section 6.0. Requirements for instrument performance are listed in Section 6.0.

4.0 DOCUMENT CONTROL PROCEDURES

Document control will be provided by the TRW Document Control Officer, M. K. O'Rell. He will be responsible for maintaining a permanent file of documents prepared during the course of the project. Each document will be assigned a document number using the suggested identification code provided by the Prime Contractor. All documents will bear the code LC 1-619-026-31 followed by a document code. The document codes to be used are found in Table 4-1 as provided by the Prime Contractor. Each document in a given code will also be assigned a serial number.

TABLE 4-1

DOCUMENT CODES

Document	Code Letter
Project Work Plans	A
Project Logbooks	B
Laboratory Notebooks	G
Laboratory Data Sheets	H1, H2 etc.
Laboratory Coding Form	J1, J2 etc.
Sample Logs	L1, L2 etc.
Internal Memos	M
External Written Communication	N
Confidential Information	O
QA Plan	Q
Reports	R
Final Report	FR
Miscellaneous	X

Copies of all the project documents will be retained in a permanent project file at TRW. These include chain of custody forms, laboratory notebooks, laboratory sample travelers, laboratory data sheets, analytical data sheets, internal memos and external written communication. Notebook entries showing chain of custody throughout the entire analytical activities will be contained in files.

5.0 CHAIN OF CUSTODY PROCEDURES

Chain of custody procedures will be used to document the identity of a sample and its handling from time of receipt at TRW until its ultimate disposal. Responsibility for this activity will reside with R. J. Lindgren, Sample Bank Manager. All samples will be inspected upon receipt to ensure that 1) shipping containers have not been opened in transit, 2) samples are properly labeled and chain of custody records agree with individual sample tags and 3) samples were not damaged in shipment. The chain of custody sheets will be signed after all comments are made by the Sample Bank Manager, and held by the Document Control Officer until directions are received from the Prime Contractor. The water samples will be stored in a locked refrigerator in a secured building (the building is limited access, controlled by security at all times). Transfer of custody of the samples to the analysis laboratory will be recorded in the sample logbook by the Sample Bank Manager. The analyst receiving the samples will also record their receipt in the analysis notebook. For the semi-volatile and pesticide samples, the extraction laboratory will maintain custody of the extracts until released to the appropriate instrumental laboratory. Transfer of custody will again be noted in the sample extraction notebook and the instrumental laboratory notebook. The instrumental laboratories will retain custody of the samples (in a locked laboratory again in the secured building) until analysis is complete. The remaining sample extracts will be returned to the Sample Bank Manager for final disposition. For the VOA samples, the samples will be transferred directly to the appropriate instrumental laboratory and recorded in each laboratory notebook.

To prevent possible mis-numbering of the samples, the GCA assigned sample bank numbers will be used throughout the analytical activities conducted at TRW. All other sample numbers (e.g., blanks) generated in the laboratory will carry the prefix LC.

Any problems or questions concerning chain of custody will be resolved by contacting the GCA QA Manager, Rosemary Ellersick. Appropriate remarks will be placed in the custody record, initialed and dated.

6.0 ANALYTICAL INTERNAL QC CHECKS

Specific quality control procedures for each of the analytical methods are presented below. All of the analytical methods employ the following quality control procedures:

- Methods Blanks
- Replicate Analyses
- Surrogates
- Laboratory Control Standards.

6.1 VOLATILE ORGANIC ANALYSIS (VOA) QUALITY CONTROL PROCEDURES

Quality control procedures for VOA analysis are described below. Included are the requirements for instrument calibration and the frequency and type of internal QC samples. Each activity is described with reference to standard methods where appropriate. In addition, a summary of the frequency, control limits, and corrective action is given.

6.1.1 Tuning of the Mass Spectrometer

Daily measurements of the GC/MS standard reference compound 4-bromofluorobenzene (BFB) will be made. The procedure for BFB involves injection of 50 nanograms of 4-bromofluorobenzene into the gas chromatograph with the following acceptance criteria applying:

Mass	Ion Abundance Criteria
50	15-40% of the base peak
75	30-60% of the base peak
95	base peak, 100% relative abundance
96	5-9% of the base peak
173	less than 1% of the base peak

174	greater than 50% of the base peak
175	5-9% of mass 174
176	greater than 50% of the base peak
176	greater than 94% but less than 100% of 174
177	5-9% of mass 176

The specified criteria will be met prior to the analysis of samples of each analysis day. This data will be recorded and provided to the Prime Contractor as a part of the deliverable data package.

6.1.2 Calibration of GC/MS System

The GC/MS system is initially calibrated to determine response factors using standards described in Section 6 of Method 624. After the system is calibrated, the calibration for organic compounds will be verified at least once each day of running (8 hours) by the use of standards containing the internal standards. If the deviation is greater than 20 percent, the system will be recalibrated. Information regarding the verification of calibration will be recorded and provided to the Prime Contractor as a part of the deliverable package.

6.1.3 Purging Efficiency of Acrolein and Acrylonitrile

The purging efficiency of the system will be determined each day for analysis of acrolein and acrylonitrile by Method 603. A mixed standard of these two compounds will be analyzed each day prior to analysis of these compounds when Method 603 is required. Control limits for recovery are as follows:

Compound	Control Limit
Acrolein	80 - 90%
Acrylonitrile	93 - 103%

6.1.4 Internal Standards

For the analysis of volatile organic compounds in water by Method 624, internal standards will be used.

6.1.5 Surrogate Compounds

Surrogate organic compounds will be added at a known concentration of about 25 micrograms per liter to every environmental sample. Surrogate compounds for volatiles in water and acceptable control limits expressed as percentage of true value are as follows:

Compound	Control Limit
Fluorobenzene	81 - 107%
p-Bromofluorobenzene	88 - 118%

6.1.6 Laboratory Control Standards

Laboratory control standards (LCS) will be employed to identify systematic method and operator errors. The LCS is a mixture of a number of compounds of known concentration spiked into reagent water. They are prepared from standards supplied by TRW. A minimum of the compounds to be used to make up the LCS for volatile organic analyses is outlined below. For Method 624 there must be one LCS processed through the entire analytical method for each set of samples processed in a group on the same day. Normally this will be one LCS for every 5 to 15 samples. A control chart will be maintained for each of the compounds with the control limits below (calculated as percent of true value) demonstrating acceptable performance.

The following standard mixture will be used (availability permitting) as a laboratory control standard for purgeables (Method 624). The LCS will contain these compounds in the range of 5-25 μg/L:

	Compound	Control Limits
Method 624:	Benzene	79 - 107%
	Chloroform	90 - 100%
	Bromoform	76 - 107%
	sym-Tetrachloroethane	67 - 110%
	p-Bromofluorobenzene	88 - 118%
	Carbon tetrachloride	85 - 102%
	Fluorobenzene	81 - 107%

Trichloroethylene	93 - 105%
Tetrachloroethylene	93 - 120%
Chlorobenzene	69 - 107%

6.1.7 Method Blanks

One method blank (reagent water) will be processed through the entire analytical procedure for every set of samples processed in a group at the same time on the same day. At least one method blank will be run per work shift. The method blank will not show any significant signals that correspond to compounds on the Love Canal or priority pollutants lists. If common laboratory contaminants are found in the method blanks, reported values for the same compounds in the environmental samples will be reduced by the amount found in the blank.

6.1.8 Replicate Analyses

Intralaboratory precision of the environmental measurements will be evaluated by the measurement of one duplicate (split) of every 10 environmental samples. A duplicate is a second aliquot of a sample that is carried through the entire laboratory analytical method. These results will be reported as part of the data base on the Love Canal Study.

6.2 QUALITY CONTROL PROCEDURES FOR SEMIVOLATILE ANALYSIS

Quality control procedures to be employed for the analysis of semivolatile organics in water are described below. Included are procedures specific to the analysis of benzidine and hexachlorocyclopentadiene. Requirements for instrument calibration and the frequency and types of internal QC samples to be run are discussed.

6.2.1 Tuning of Mass Spectrometer

Daily measurements of the GC/MS standard reference compound decafluorotriphenylphosphine (DFTPP) will be made. The procedures and criteria to be met for DFTPP are as set forth in Method 625. These criteria must be met prior to the analysis of samples each analysis day. This data will be recorded and provided to the Prime Contractor.

6.2.2 Calibration of GC/MS System

The GC/MS system must be initially calibrated to determine response factors using standards as described in Section 6 of Method 625. After the system has been calibrated, the calibration for organic compounds must be verified at least once each day of running (8 hours) by the use of standards containing the internal standards. If the deviation is greater than 20 percent, the system will be recalibrated. Information regarding the verification of calibration will be provided to the Prime Contractor.

6.2.3 Internal Standards

Internal standards are added to each sample according to the procedures outlined in Section 6.4 of Method 625. The compounds used as internal standards for the analysis of semivolatile organics in water are the following:

anthracene d_{10}
naphthalene d_8
chrysene d_{12}

6.2.4 Surrogate Compounds

Surrogate organic compounds will be added at a known concentration of 5 - 25 µg/L to every environmental sample. The analytical performance, expressed as a percentage of the true values for these compounds, will be monitored with the control limits given below indicating acceptable performance. The surrogate compounds for semivolatiles in water are the following:

Compound	Control Limits
2-fluorophenol	18 - 58%
1-fluoronaphthalene	33 - 88%
4,4'-dibromooctafluorobiphenyl	34 - 98%

6.2.5 Laboratory Control Standards (LCS)

Laboratory Control standards (LCS) will be employed to identify systematic method and operator errors. The LCS is a mixture of a number of compounds of known concentration spiked into reagent water. A minimum of the compounds to be used to make up the LCS for semivolatile organic analyses are given below. One LCS will be processed through the entire analytical method for each set of samples extracted in a group at the same time on the same day. Normally there will be one LCS for every 5 - 15 samples depending on how many samples are extracted in a given batch.

Standards for base/neutrals and acids LCS will be prepared at a concentration in the range of 5 - 25 μg/L using standards supplied by TRW. The minimum (dependent on availability) standard compounds to be used and control limits expressed as percentage of true values are as follows:

Compound	Control Limits
1,4-dichlorobenzene	16 - 64%
1,2,4-trichlorobenzene	36 - 96%
1,2,3,4-tetrachlorobenzene	35 - 59%
Fluoranthene	40 - 118%
2,4,6-trichlorophenol	46 - 82%
2-chloronaphthalene	34 - 98%
2,6-dinitrotoluene	34 - 94%
4-nitrophenol	36 - 68%
2-fluorophenol	18 - 58%
Pentachlorophenol	63 - 111%
Beta-benzenehexachloride	19 - 119%
1-fluoronaphthalene	33 - 81%
4,4-dibromobiphenyl	36 - 94%
4,4-dibromooctafluorobiphenyl	34 - 98%
di-n-butylphthalate	45 - 125%

A control chart will be maintained for each of the analytes.

6.2.6 Method Blanks

One method blank (reagent water) will be processed through the entire laboratory analytical procedure for each set of samples extracted in a group at the same time on the same day. Normally there will be one method blank for every 5 to 15 samples depending on how many samples are extracted in a given batch. The method blank must not show any significant signals that correspond to compounds on the Love Canal or priority pollutants list. If common laboratory contaminants, e.g., phthalate esters, are found in the method blanks, reported values for the same compounds in the environmental samples will be reduced by the amount found in the blank.

6.2.7 Replicate Analyses

Intralaboratory precision of the environmental measurements will be evaluated by the measurement of one duplicate (split) of every 10 environmental samples. Due to the lack of sufficient sample for splitting the duplicate,analyses will be performed on the extracts, duplicating only the analytical finish of the method.

6.2.8 Capillary Column Performance

The performance of the capillary chromatographic column used will be verified daily before analysis by injection of known mixtures. A procedure for this performance test is as follows:

- 100 ng pentachlorophenol and benzidine simultaneously using a 1.0 μL injection.
- Analysis of the Grob mixture.
- Criterion: Tailing factor ±1.5 for these compounds.

6.3 QUALITY CONTROL PROCEDURES FOR PESTICIDE ANALYSIS

The quality control procedures to be utilized in these analyses are those described in Method 608. Calibration and standardization procedures for the gas chromatograph are sufficiently described in Section 6 of Method 608 and will not be further discussed in this section. Requirements for analysis of method blanks, replicates, and known QC samples are described below.

6.3.1 Method Blanks

One method blank (reagent water) will be processed through the entire laboratory analytical procedure for each set of samples extracted in a group at the same time on the same day. Normally this will be one method blank for every 5 to 15 samples, depending on how many samples are extracted in a given batch. The method blank must not show any significant signals that correspond to compounds on the Love Canal or priority pollutants list. If common laboratory contaminants e.g., phthalate esters, are found in the method blanks, reported values for the same compounds in the environmental samples must be reduced by the amount found in the blank.

6.3.2 Laboratory Control Standards (LCS)

Laboratory control standards (LCS) will be employed to identify systematic method and operator errors. The LCS is a concentrate of one or more compounds of known concentration spiked into reagent water.

Presently, the LCS for pesticides in water will consist of (at a minimum) 5 - 25 μg/L of Aroclor 1242. Control limits for this compound have not yet been established. One LCS must be processed through the entire analytical method for each set of samples extracted in a group at the same time on the same day. Normally there will be one LCS for every 5 to 15 samples, depending on how many samples are extracted in a given batch. Results of LCS analysis are to be recorded on the appropriate data sheets and must include measured and expected concentration for each component of the LCS.

6.3.3 Replicate Analyses

Intralaboratory precision of the environmental measurements will be evaluated by the measurement of one duplicate (split) of every 10 environmental samples. A duplicate is a second aliquot of a sample that is carried through the entire laboratory analytical method and is dependent on available sample.

7.0 DATA REPORTING MANAGEMENT SYSTEM

Data reporting will be to the Prime Contractor on the provided standardized forms. Initial data review will be provided by the responsible individual for each of the analytical areas as previously shown in the Project Organization Chart (Figure 1-1). Additionally all data will be reviewed by the Project Manager prior to disbursement to the Prime Contractor.

8.0 CORRECTIVE ACTION PROCEDURES

Corrective actions as specified in Section 6.0 will be implemented by the responsible work package manager as specified in Figure 1-1. The required corrective actions will be documented and implementation will be verified by the Project Manager.

Appendix Q
Plan 4.8

Acurex Corporation

Revised QC Plan

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SECTION 1

PROJECT DESCRIPTION AND ORGANIZATION

GCA/Technology Division is currently under contract to the Environmental Protection Agency (EPA) to evaluate pollutant levels present in the Love Canal area of Niagra Falls, New York. This program involves a multifaceted sampling and analytical effort designed to detect and quantify a variety of trace metals, volatile organic compounds (VOA's), pesticides, and other compounds in soil, sediment, air, biota, and water samples.

To expedite the program in a timely manner, GCA will be conducting the analytical efforts through Acurex Corporation (lab code no. 26) and other subcontractors. For this project, Acurex has set up the following management organization:

- Laboratory Director -- John Taylor
- Quality Assurance (QA) Coordinator -- Erick Hagmann
- Contract Lead Chemist (Internal QA/QC and Corrective Action) -- Dr. William Fitch
- Document Control and Chain of Custody -- Linda Bohannon

SECTION 2

MAJOR PROJECT ELEMENTS

The scope of work consists of the three tasks discussed in this section.

2.1 WATER SAMPLES -- ORGANIC COMPOUNDS (RFP TASK III)

Acurex shall analyze a number not to exceed 150 water samples by selected procedures. In completion of this work, Acurex shall:

- Analyze a number not to exceed 150 sample fractions for VOA's by Method 624 (RFP subtask 1)
- Analyze a number not to exceed 15 sample fractions for semivolatiles by Method D for water, at the direction of the technical monitor (RFP subtask 5)
- Analyze a number not to exceed 150 sample fractions for pesticides and related compounds by the method for Pesticides in Water (RFP subtask 6)
- Whenever acrolein and/or acrylonitrile is detected in the performance of the volatile organic analysis, or if directed by the technical monitor, quantify acrolein and/or acrylonitrile in accordance with the Acrolein and Acrylonitrile Quantitation Procedure. The number of such quantitations shall not exceed 35 (RFP subtask 7).

- Whenever benzidine is detected in the performance of the semivolatile analysis, or if directed by the technical monitor, quantify benzidine in accordance with the Benzidene Quantitation Procedure. The number of such quantitations shall not exceed 35 (RFP subtask 8).
- Perform qualitative identifications by the GC/MS Qualitative Analysis Procedure on a number not to exceed 50 samples, as directed by the technical monitor (RFP subtask 10)

2.2 SOILS AND SEDIMENTS -- SEMIVOLATILES METHODS STUDY (RFP TASK IV)

Acurex shall analyze a number not to exceed 25 soil and sediment samples by capillary GC/MS after preparation by the three methods for semivoltatiles. The anticipated split between soil and sediment samples is 20 and 5, respectively. Based on the data resulting from these analyses, a single method will be selected by EPA. The data generated from the analysis of the original 25 samples shall be reported concurrently to GCA and to designated EPA personnel within 30 calendar days of receipt of the samples.

For the selected method, Acurex shall analyze 7 of the original 25 samples after spiking. All spiking materials will be provided by GCA. The percent recovery of the compounds shall be calculated from these data. The recovery data shall be reported within 14 days after notification of the selected method.

2.3 SOIL AND SEDIMENT SAMPLES -- ORGANIC COMPOUNDS (RFP TASK V)

Acurex shall analyze soil and sediment samples by selected procedures. In completion of this work, Acurex will:

- Analyze a number not to exceed 140 sample fractions for VOA's by modified Method 624 (RFP subtask 1)

- Analyze a number not to exceed 65 sample fractions for pesticides and related compounds by the Pesticides in Soils Method (RFP subtask 10)
- Analyze a number not to exceed five sample fractions for pesticides and related compounds by the Pesticides in Sediments Method (RFP subtask 11)
- Whenever benzidine is detected in the performance of the semivolatile analysis, or if directed to do so by the technical monitor, Acurex shall quantitate benzidine in accordance with the Benzidine Quantitation Procedure. The number of such quantitations shall not exceed 15 (RFP subtask 12).
- Perform qualitative identifications by the GC/MS Qualitative Analysis procedures on a number not to exceed 20 samples, as directed by the technical monitor

The definition of a sample, as referred to herein, is that collection of sample fractions necessary to complete the analyses specified above submitted to Acurex with unknown organic content.

SECTION 3

QUALITY ASSURANCE OBJECTIVES

Precision and accuracy must be discussed for each individual compound analyzed by each method. The following numbers are averages. For example, polar phenols such as dinitrophenol are much less reliably analyzed than are nonpolar compounds such as naphthalene.

PRECISION AND ACCURACY

	VOA's	Semivolatiles	Pesticides
Soil	±30 percent	±100 percent	±50 percent
Water	±20 percent	±50 percent	±30 percent

SECTION 4

DOCUMENT CONTROL PROCEDURES

The purpose of document control is to ensure that all project documents will be accounted for when the project is complete. Table 4-1 lists the principal subcontractor items subject to document control with records maintained by the person listed as document control officer (DCO) for that document. Table 4-2 gives a more complete listing of documents generated during the project.

Table 4-1 identifies a DCO for each operation who is responsible for issuing, controlling, and maintaining, records of controlled documents.

Table 4-1. Document Control Officers

Item	Issued By	Interim Responsibility	DCO
Written communication	Various people	Recipient	Deanna Lamphere
Analytical sample tags	Sample bank	Shipper	Linda Bohannon
Laboratory notebooks	Subcontractor	Analytical subcontractor	Bill Fitch and Greg Nicoll
Analytical data sheets	GCA	Analytical subcontractor	Linda Bohannon
QA project plan	Subcontractor	Subcontractor	Erick Hagmann

Table 4-2. Project Documents

- Project work plans
- Project logbooks
- Sampling logbooks
- Sampling data sheets
- Sampling coding form
- Laboratory notebooks
- Laboratory data sheets
- Laboratory coding form
- Sample logs
- Internal memos
- External written communication
- Confidential information
- Photos, maps, and drawings
- QA plan
- Reports
- Final report
- Miscellaneous

SECTION 5

CHAIN OF CUSTODY

The procedures identified here have been established for this project by the GCA QA manager with NEIC staff members and following guidelines developed from the following two publications:

- NEIC Policies and Procedures Manual, EPA-330/9-78-001R, Section II
- Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, NEIC, April 1980, Sections VIII and IX

The purpose of chain-of-custody procedures is to document the identity of the sample, and its handling, from its first existence as a sample until its ultimate disposal. It must be possible, via a custody record, to trace a sample through all transfers of custody and to verify that it is the same sample collected.

To establish that a sample is valid, it is also necessary to document that no tampering has occurred -- either to the sample itself, the sampling equipment used, or the environment sampled. This is done by the use of evidence tapes, locks and custody seals, and documented entries noting their condition in field and laboratory log books. The custody record must also document any tampering that occurred; the absence of any such comments indicates no tampering during the period of custody.

The GCA sample bank will provide and dispense evidence tapes, sample tags, custody records, logbooks, field and laboratory data sheets, custody seals, and locks as appropriate. The responsibility at Acurex for ensuring that custody procedures are followed belongs to Linda Bohannon.

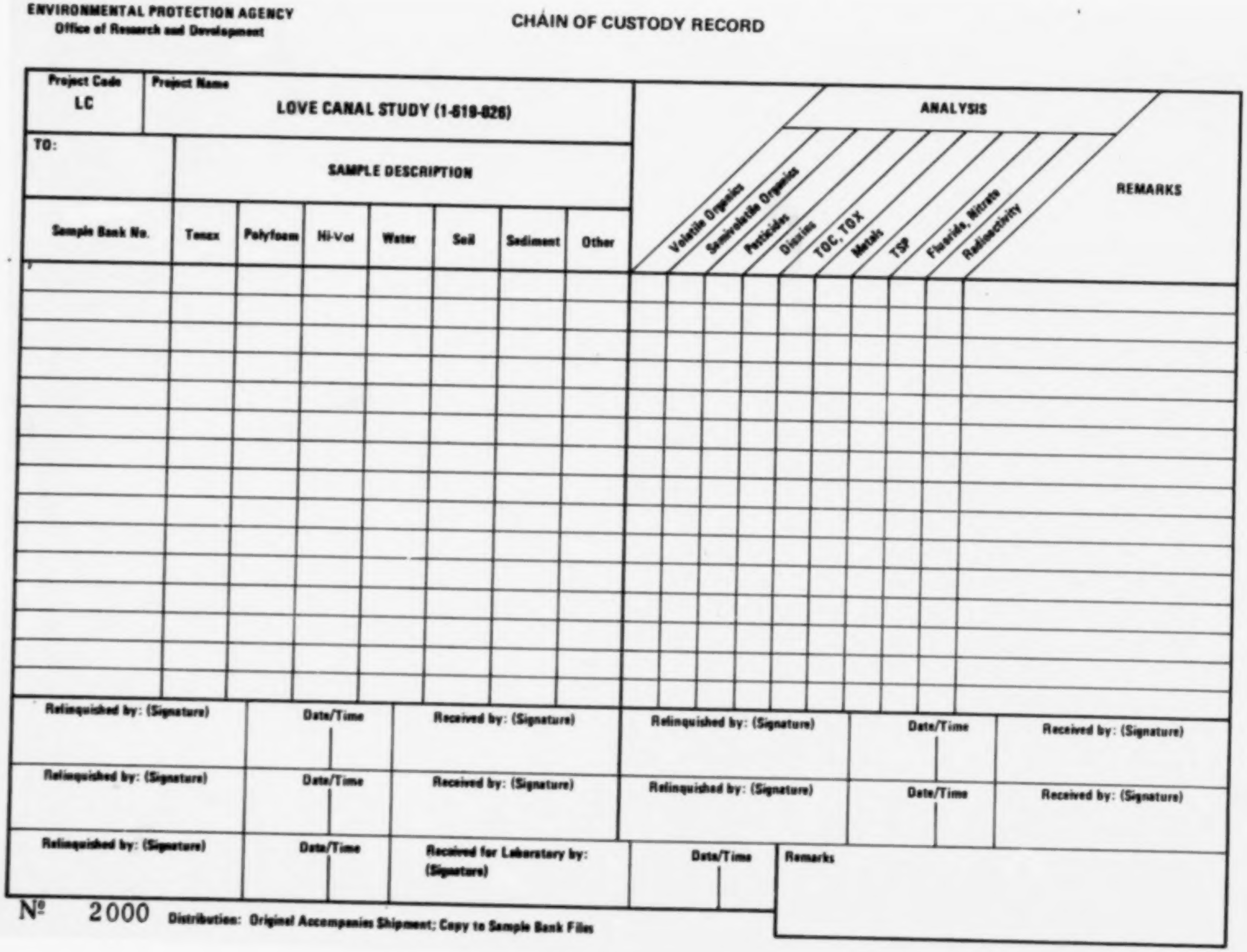
Customized record sheets, following the NEIC format, have been provided for this program. Figure 5-1 shows the custody record used for all shipping of samples to analytical laboratories.

If a discrepancy between sample tag numbers and custody record listings is found, the person receiving custody should document this and properly store the samples. The samples should not be analyzed until the problem is resolved by contacting the GCA QA manager or other designated responsible authority.

The responsible person receiving custody should attempt to resolve the problem by checking all available information (other markings on sample container, type of sample, etc.). he should then document the situation on the custody record and in his project logbook and notify the GCA QA manager by the fastest available means, followed by written notification.

Changes may be written in the "Remarks" section of the custody record and should be initialed and dated. A copy of this record should accompany the written notification to the QA manager.

The sample bank, the sampling subcontractor's laboratory area, and each subcontracting analytical laboratory are considered to be working laboratories. It is assumed that the laboratory is not open to the general public but has access restricted to employees known to each other. Each handling of the sample to renumber, subdivide, preserve, etc., does not have to be documented in this situation.



ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development

CHAIN OF CUSTODY RECORD

Project Code LC

Project Name LOVE CANAL STUDY (1-619-026)

TO:

	SAMPLE DESCRIPTION							ANALYSIS									
Sample Bank No.	Tenax	Polyfoam	Hi-Vol	Water	Soil	Sediment	Other	Volatile Organics	Semivolatile Organics	Pesticides	Dioxins	TOC, TOX	Metals	TSP	Fluoride, Nitrate	Radioactivity	REMARKS

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks	

№ 2000 **Distribution: Original Accompanies Shipment; Copy to Sample Bank Files**

Figure 5-1. Chain-of-Custody Record

SECTION 6

ANALYTICAL INTERNAL QUALITY CONTROL CHECKS

6.1 QUALITY ASSURANCE ORGANIZATION

The Acurex Analytical Laboratory QA coordinator (Erick Hagmann) will coordinate and review the development and implementation of the quality control (QC) procedures to be used for laboratory programs. The contract lead chemist (Bill Fitch) will be responsible for development and implementation of all aspects of quality control within this program. He will prepare the necessary forms, QC schedules, and reporting formats. He will interface with the analysts and project management on a daily basis on QA problems and progress.

6.2 QUALITY CONTROL REPORTING AND FEEDBACK

A QC form will be used for each of the analytical tasks in a program. Acceptance criteria for agreement between analytical results and reported values will be incorporated on the forms. The additional QC checks (acceptable retention time, minimum standard areas, minimum resolution, and agreement of repeated standards) will remain as part of the laboratory's basic QC effort.

These QC forms will be filled out by the analytical personnel daily as the data is generated. They will be reviewed each day by the project lead chemist. Any problem areas or trends will be noted with copies of the forms going back to the appropriate laboratory the next day. This system will help correct problems as they occur with immediate feedback to the analysts and project management.

6.3 QUALITY CONTROL FOR EQUIPMENT, REAGENTS, AND SUPPLIES

The laboratory approach to QC of equipment, reagents, and supplies is to obtain high-quality items for all analytical work. Up-to-date equipment manuals are to be made available and instrument notebooks are to be kept for each actual piece of equipment.

Reagents, solvents, and standards with specific levels of purity will be specified for each type of analysis. Each new solvent lot will be analyzed for contaminants. Specifications for GC column materials, glassware, and sample handling equipment will also be determined.

6.4 ANALYTICAL QUALITY CONTROL

Specific QC analyses will be made for each type of analysis to determine and document current levels of recoveries, interferences, precision, and accuracy. The need for a stringent, continual QC effort is exemplified by the potential problems which can occur with the purge and trap technique. These problems include:

- Incomplete purging
- Breakthrough if the purging time is too long
- Systems with solenoid valves and long lengths of transfer tubing may suffer "memory" effects from previous samples
- Traps packed with Tenax and silica gel can deteriorate and "steam clean" the chromatographic column during the thermal desorption step
- Traps packed only with Tenax to avoid water interferences may allow breakthrough of some compounds

- Heated stainless steel transfer lines can cause decomposition of some compounds
- Stock solutions in methanol, at concentrations greater than 1,000 μg/l must be used to prepare standards at concentrations of 1 μg/l or less in water to achieve reproducibility at low concentrations

A specific schedule of QC analysis will be developed for each type of analysis. This schedule will include:

- A blank sample with each sample set extracted or purged
- A set-in standard that meets minimum criteria
- Repeat samples to document analytical reproducibility
- Unknown check samples or QA standards to document accuracy
- Spikes with known concentrations added to each sample to measure extraction recovery and purging efficiencies

Minimum levels for agreement between repeated results and agreement with known standards will be incorporated from the contract. Experience has shown that the precision of this duplicate data depends on the concentrations of the compounds and the period of time (days, weeks, or months) between analyses. Concentrations near the detection levels often show wide variations between repeat analyses. Any data outside of established limits for any of the QC parameters will cause analyses to be stopped temporarily until the problem is found and corrected.

SECTION 7

DATA REPORTING MANAGEMENT SYSTEM

An integral part of all programs involving the analytical laboratory is a comprehensive data management system. The goal of this system is to document and insure acceptable levels of accuracy and precision from the analytical system.

In developing a QA program, all aspects of the laboratory activities are reviewed as they relate to needs for QC. These aspects include:

- Organization of QA function
- Recordkeeping and sample control
- QC reporting and feedback systems
- Reagents and supplies
- Instrument calibration and standards
- Quality control of the analytical system
- Data validation and review
- Quality assurance audits

7.1 ORGANIZATION OF QUALITY ASSURANCE FUNCTIONS

The QA coordinator (Erick Hagmann) coordinates and reviews the development and implementation of the specific QC activities. He then reviews these activities on a regular basis and schedules independent audits as needed.

The contract lead chemist (Bill Fitch) is responsible for the following: developing necessary QA documents, scheduling QA activities, and coordinating the reporting, recordkeeping and QC data analysis. He is responsible for maintaining mechanisms for problem detection, reporting, and correction.

The lead chemist can become aware of problem areas and shifting priorities as they arise. The independent review and involvement of the QA coordinator is available to the program as needs are perceived by the technical staff, management or by the program clients.

7.2 RECORDKEEPING AND SAMPLE CONTROL

Detailed recordkeeping and control of samples are essential to a successful laboratory program. A basic requirement of the QA program is a procedure for recording and logging all samples received and handled. An inventory system effective in controlling the disposition of samples and standards is in place at Acurex.

Other aspects of QC recordkeeping requirements include:

- Up-to-date analytical manuals
- Instrument logs
- Specific lab notebooks for each program task
- Formal recordkeeping procedures for all QC data including calibrations, blank analyses, standards analyses, and duplicate samples

Sample control in the laboratory begins the day the samples are received. When large numbers of samples are involved, separate log books are established for use only with those samples. As each sample is received it is assigned a unique number and is logged into the master log book. This log includes date of receipt, sample description (from label), sample type (i.e., amber gallon jug water, sealed 5-ml ampule, etc.), and analyses to be performed. A sample sheet is prepared including such additional information as special handling instructions, extraction procedures, cleanup and/or other special preparation needs, and route (i.e., Extractions Laboratory, GC Laboratory, GC/MS Laboratory, etc.). This is carried with the sample throughout its life and is attached to the final data output. An example of this form is shown in figure 7-1.

A copy of each sample sheet is sent to the master file. When all analyses are completed the sample sheet is returned to the master file. Each person working with a sample maintains a bound notebook where all work done on a sample is recorded. Thus, the complete history of a sample is maintained.

7.3 QUALITY CONTROL FOR REAGENTS AND SUPPLIES

Most analytical procedures have detailed requirements for reagents and supplies. Reagents, solvents, and standards with specific levels of purity are usually specified in the test procedure. Specific GC column materials, glassware, and sample handling equipment will also be specified. The QC procedures for equipment and supplies typically include the following items:

- Operator checklists for required supplies
- Documentation and reporting of all deviations from specified equipment
- Procedures for testing for purity of reagents
- Tolerances for glassware

7.4 INSTRUMENT CALIBRATION AND STANDARDS

The quality of all test results can be directly related to proper calibration procedures. As part of any QC program, calibration procedures

DEPARTMENTS: GEN / ELEM / EXT / GC & GC/MS /________ LAB ID NO. ____________________

DATE RECEIVED______________ RESULTS PROMISED __________ LOCATION ____________

CLIENT (PROJECT)________________________________ PROJ. NO.__________

ADDRESS________________________________ CLIENT P.O.__________

________________________________ QUOTED COST__________

ATTN ________________________________

PHONE ________________________________

NO. OF SAMPLES __________ TAKEN BY: THEM / US

TYPE: D / W / ________________________ BROUGHT BY: THEM / US / FREIGHT

RETURN / STORE: DURATION________________ W / M / Q / S-A / A

SUB-CONTRACT: DATE______________ P.O. NO.______________ TO______________

DATE______________ P.O. NO.______________ TO______________

DASH	SAMPLE IDENTIFICATION	ANALYSIS REQUIRED	GEN	ELEM	EXT	GC/MS	OTHER

Figure 7-1. Acurex Traveller

and standards are specified for all equipment and supplies used in the analytical procedures. Traceability to primary standards is essential for test procedures to be used in multiple laboratories. Quality assurance procedures for standards and calibration typically include the following considerations:

- Written, detailed calibration instructions
- Preparation procedures for secondary standards
- Requirements for frequency of calibration
- Recordkeeping and reporting of all calibrations and standards used
- Evaluation of internal standards
- Tolerances for calibration requirements

7.5 QUALITY CONTROL OF THE ANALYTICAL SYSTEM

Routine QC samples to be analyzed concurrently with samples is often the most significant portion of laboratory QA program. The purpose of these checks are twofold: (1) to assure that samples being analyzed satisfy predetermined standards of accuracy, and (2) to measure and document occurring levels of accuracy and precision. There are many different types of QA samples which could be used for these purposes. The correct combination of these depend on the economics and complexity of the analytical procedure and the desired degree of accuracy. The following QC parameters are considered for appropriateness to a particular analytical method and particular program.

7.5.1 Interferences in the Analytical Systems

The analytical results of a test method might be affected by interferences in the glassware, solvents, reagents, or in the sample matrix. Blank samples subjected to conditions similar to samples being analyzed can be used to evaluate the effect of interferences on the analyses. Specific cleanup procedures can be used to minimize interferences from the sample matrix. The frequency of blank analysis depends on the findings from previous analyses which are charted and continually reviewed. For example, experience has shown that it is an absolute necessity to analyze blank samples for purgeable organic samples. If the purge and trap system being used retains "memory" from previous analyses, it may increase the values actually present in water samples.

7.5.2 Extraction Recovery and Purging Efficiency

The accuracy of the analytical test data is directly related to the extraction or purging recovery. Recovery is measured using surrogate standards spiked into each sample.

7.5.3 Analytical Accuracy and Specificity

Accuracy of the analytical system is probably the single most important QC parameter. "Accuracy" is a measure of how well the analytical data agrees with a "true" value. Accuracy is affected by recoveries, interferences, calibration, and any other biases introduced in the sample analysis. Accuracy can also be affected by problems in qualitative specificity. In complex sample matrices with multiple compounds, the use of retention times can lead to misidentification of compounds. The extent of misidentification can be estimated by repeated analysis of standards containing compounds of interest. One way to best quantify the accuracy of the entire analytical system is through the use of blind standards. Acurex analyzes "QA standards" on a regular basis.

7.6 QUALITY ASSURANCE AUDITS

In addition to the daily review of QC data, regular system audits and data quality assessments will be a part of Acurex QA systems. The contract lead chemist for the project may conduct these reviews or they may be done independently by the QA coordinator at the request of program management or client. Performance audits by independent groups reporting to clients may also be done on certain programs.

Audit checklists will be prepared for some programs. Any weak points in the QC protocol will be reported back to the QA coordinator for correction. Data quality assessments usually involve a review of QC data. QC parameters such as analytical precision, recovery levels, and accuracy are reestimated based on current data and compared with past levels.

SECTION 8

CORRECTIVE ACTION PROCEDURES

Troubleshooting procedures that will be applied to programs have been developed through experiences with other similar programs. Basically, these procedures involve methods for identifying that a problem exists, identifying the cause of the problem, and arriving at a solution to the problem. The procedures identified for fulfilling effective troubleshooting are described in the following paragraphs.

8.1 IDENTIFYING THE EXISTENCE OF PROBLEMS

The three major sources of information that will be used on this program to indicate the existence of an analysis problem are as follows:

- In-house QA programs
- Observations by technical personnel
- Replicate analysis data with poor agreement between results

The in-house QA programs will be the primary method used to identify problems that arise in the analytical schemes for the samples generated under most contracts (through the use of spiked samples, standards, and blank samples). These procedures will allow detection of problems associated with all aspects of the schemes.

Observations by technical personnel during the extraction, sample preparation, and analyses are also an important avenue for recognizing problems, especially where random errors or malfunctions are concerned.

All laboratory personnel are encouraged to report all suspected abnormalities in each operation concerned with sample handling. The extensive experience of Acurex personnel in handling and analyzing environmental samples and their resultant insight means that this is a very effective method for identifying and controlling problems.

Comparison of the results of replicate analyses constitutes a third source of information that is used to indicate the existence of a problem. Significant variations between replicate analyses or long-term trends of analytical results indicate the existence of a problem. When inconsistencies or trends above acceptable levels are found the responsible person(s) will be notified that a problem exists.

8.2 IDENTIFYING THE CAUSES OF PROBLEMS

Identification of the source of a problem begins at the procedural step where the problem was first detected. Each operation involved in the analytical scheme is then examined in reverse order. Each piece of equipment and procedure is tested with blank and standard samples or other appropriate methods until its proper operation is assured. This provides a logical, sequential method for tracking the source of the problem instead of random investigations of various steps or pieces of equipment.

In general, problems will be identified from analytical results. Therefore, the first step would be to double and triple check all calculations and data transfers. The next step would be to repeat the analysis to see if the problem was caused by a random error or malfunction. If a continuous problem is identified, the analytical instrument used for the analyses should be thoroughly examined for both hardware malfunction and operator error. This would involve the use of blank and standard samples. If no problem is identified, procedures and equipment in the next prior step, concentration or liquid chromatography for example, would be investigated. This step-wise procedure would be followed until the problem is found.

8.3 SOLVING THE PROBLEMS

When a problem has been identified, work will immediately begin to arrive at a solution. Although the solution will be different for each individual problem, the following are the general types of solutions that will be used:

- Repair of malfunctioning equipment
- Revision of improper procedures
- Additional training and supervision in cases of errors by personnel

The contract lead chemist and QA coordinator will be kept fully informed concerning problems identified and solutions employed.

8.4 DOCUMENTATION OF CORRECTIVE ACTION

All problems encountered are thoroughly documented and included on analytical data sheets. In addition, a record of equipment maintenance is kept in a logbook for each instrument.

Energy Resources Co., Inc. (ERCO)

QA Plan

LC 1-619-026-27-Q

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1.0 PROJECT DESCRIPTION AND ORGANIZATION

Energy Resources Co. Inc. (ERCO) has agreed to supply its analytical services to GCA Corporation, the prime contractor, to assist in a study of Love Canal. These services entail the analysis of water, soil, and sediment samples for the inorganic parameters listed in Table 1.

The water samples will be prepared according to the interim method specified by U.S. EPA (1980).[1] The concentrated digestates will be subsequently analyzed for 12 metals by inductively coupled plasma (ICP) emission spectroscopy. Mercury and selenium in water samples and all other trace metal analyses of soils and sediments will be performed by atomic absorption spectroscopy according to methods specified by U.S. EPA (1979).[2] Fluoride and nitrate analysis of the water samples will be performed according to the methodology described by U.S. EPA (1979).[3]

All sample preparations and analyses will be performed by ERCO's Trace Metals Laboratory. The management structure and key personnel involved in performance and technical quality control of analytical services for this program are shown in Figure 1.

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[1] U.S. Environmental Protection Agency. 1980. Inductively coupled plasma (ICP) atomic emission spectrometric method for trace element analysis of water and wastes. U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, July 1980.

[2] U.S. Environmental Protection Agency. 1979. Methods for chemical analysis of water and wastes. U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1979. EPA-600/4-79-020.

[3] U.S. EPA, 1979.

Table 1.—Inorganic parameters to be determined in water, soil, and sediment samples collected during the Love Canal Study

Antimony
Arsenic
Barium
Beryllium
Cadmium
Chromium
Copper
Fluoride[1]
Lead
Mercury
Nickel
Nitrate[1]
Selenium
Silver
Thallium
Zinc

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[1] To be determined in water samples only.

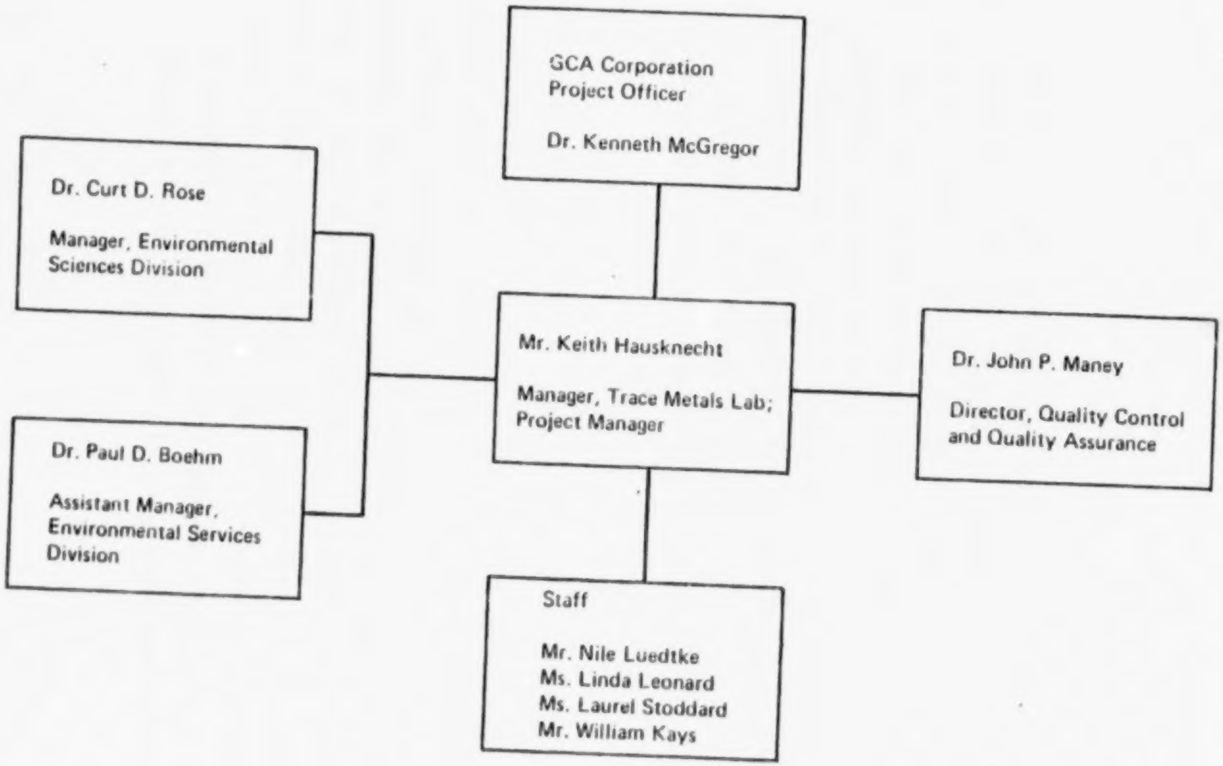
GCA Corporation
Project Officer
Dr. Kenneth McGregor
Dr. Curt D. Rose
Manager, Environmental Sciences Division
Mr. Keith Hausknecht
Manager, Trace Metals Lab; Project Manager
Dr. John P. Maney
Director, Quality Control and Quality Assurance
Dr. Paul D. Boehm
Assistant Manager, Environmental Services Division
Staff
Mr. Nile Luedtke
Ms. Linda Leonard
Ms. Laurel Stoddard
Mr. William Kays

Figure 1. Management structure and key personnel involved in performance and technical quality control of analytical services in support of the Love Canal study.

2.0 MAJOR PROJECT ELEMENTS

The project contract is subdivided into four project elements: chain-of-custody and document control, sample preparation, sample analysis, and data reporting.

The history of samples must be documented by means of an appropriate "chain-of-custody" protocol since data generated from their analysis is used as evidence in enforcement work. The possession of samples must be documented from the time the samples are collected until they are introduced as evidence in legal proceedings. For this program, customized chain-of custody records supplied by GCA will be used in conjunction with ERCO's normal sample tracking procedures to ensure complete documentation. Procedures follow those recommended by U.S. EPA/NEIC.

Once samples are properly received at ERCO's Trace Metals Laboratory, they are prepared for analysis according to methods specified by GCA Corporation in RFP #1-619-026-222-002, Appendix 1, as modified in the September 2, 1980 update. Sample preparation of water samples prior to analysis by ICP analysis (Figure 2) requires a minimum five-fold concentration of the digestate when processing the samples according to the digestion procedure for total metals.[1] Mercury analysis of water samples requires a separate digestion procedure (Figure 2).[2] A third digestion is used to prepare samples for selenium

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[1] U.S. EPA, 1980.

[2] U.S. EPA, 1979, Method 245.1.

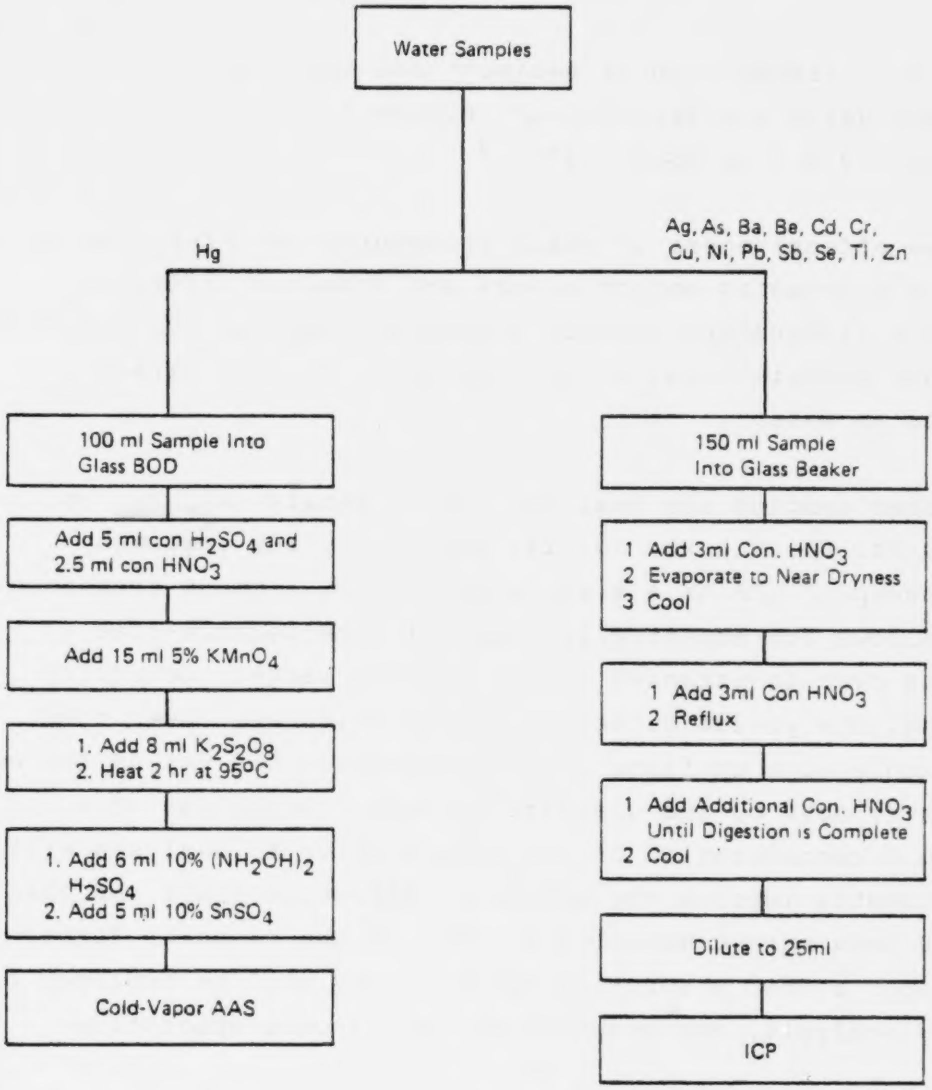
Water Samples
Hg
Ag, As, Ba, Be, Cd, Cr, Cu, Ni, Pb, Sb, Se, Tl, Zn
100 ml Sample Into Glass BOD
Add 5 ml con H_2SO_4 and 2.5 ml con HNO_3
Add 15 ml 5% $KMnO_4$
1. Add 8 ml $K_2S_2O_8$
2. Heat 2 hr at 95°C
1. Add 6 ml 10% $(NH_2OH)_2H_2SO_4$
2. Add 5 ml 10% $SnSO_4$
Cold-Vapor AAS
150 ml Sample Into Glass Beaker
1 Add 3ml Con. HNO_3
2 Evaporate to Near Dryness
3 Cool
1 Add 3ml Con HNO_3
2 Reflux
1 Add Additional Con. HNO_3 Until Digestion is Complete
2 Cool
Dilute to 25ml
ICP

Figure 2. Analytical procedures for determination of trace metals in water samples.

analysis.[1] Preparation of sediment and soil samples is performed using the methodology (Figure 3) specified by U.S. EPA (1978)[2] or USGS (1979).[3]

The effectiveness of these procedures is determined by means of processing method blanks and standard reference materials (laboratory control standards) such as NBS Standard Reference Material 1645 (River Sediment) or 1643 (Trace Elements in Water).

Water samples are analyzed for 12 metals (Ag, As, Ba, Be, Cd, Cr, Cu, Ni, Pb, Sb, Tl, and Zn) by ICP emission spectroscopy. ICP is a simultaneous multi-element technique which allows for essentially chemical interference-free analysis over an extended linear working range. Generally speaking, ICP yields detection limits which are lower than those achievable by flame atomic absorption but not as low as those available by the graphite furnace. A minimum of a five-fold concentration of the sample prior to analysis will significantly improve the practical detection limit and make the ICP data more comparable to that of the graphite furnace. Cold vapor atomic absorption spectroscopy will be employed for mercury analysis, and graphite furnace atomic absorption

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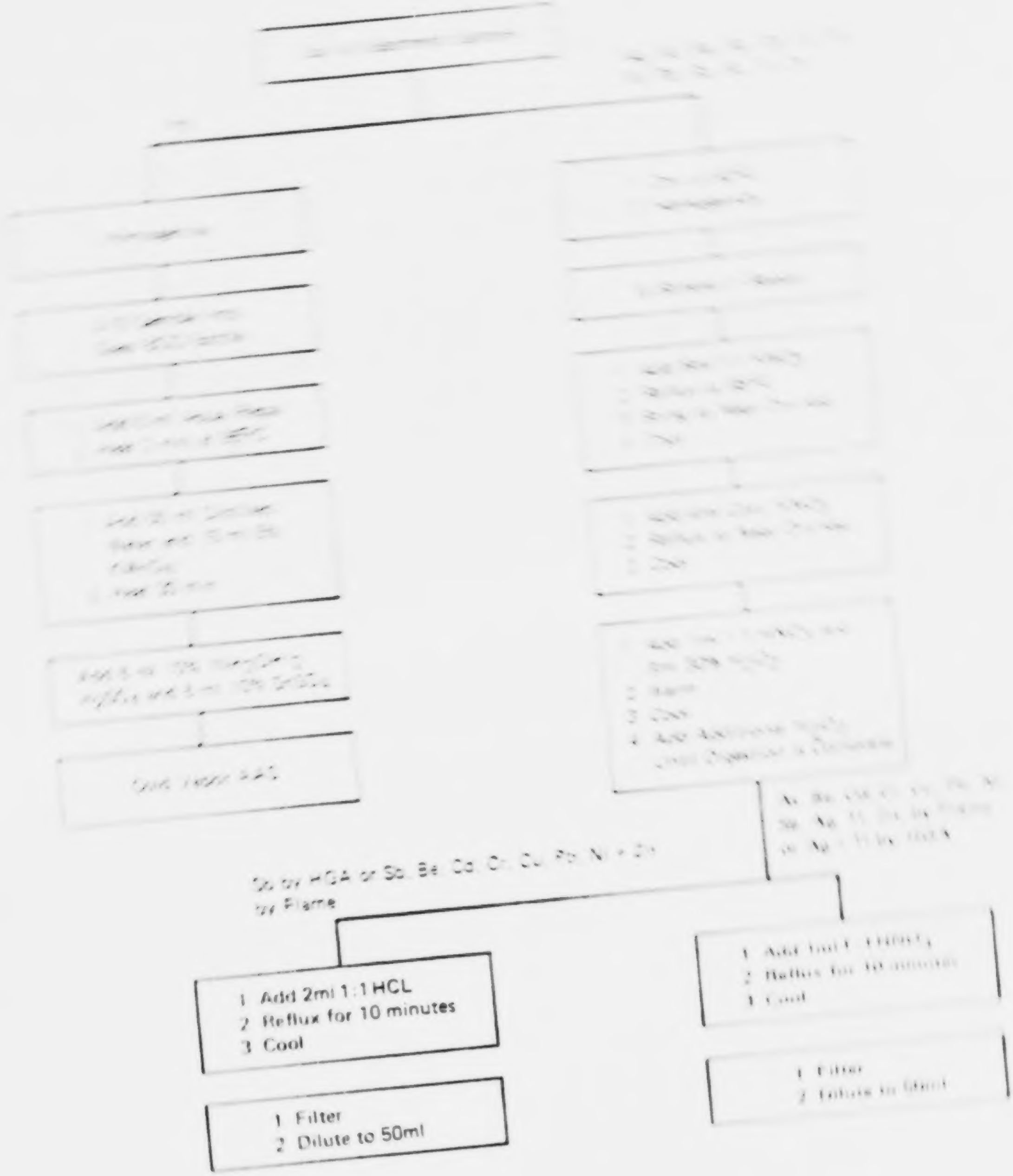
[1] U.S. EPA, 1979, Method 270.2.

[2] U.S. Environmental Protection Agency. 1978. Interim method for the analysis of elemental priority pollutants in sludge. U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, December 1978.

[3] U.S. Geological Survey. 1979. Methods for determination of inorganic substances in water and fluvial sediments. Chapter A1 in Techniques of water resources investigations of the United States Geological Survey. Washington, D.C., U.S.P.O. #024-001-03177-9.



Sb by HGA or Sb, Be, Cd, Cr, Cu, Pb, Ni + Zn
by Flame
1 Add 2ml 1:1 HCL
2 Reflux for 10 minutes
3 Cool
1 Filter
2 Dilute to 50ml
2 Reflux for 10 minutes
3 Cool
1 Filter

Figure 3. Analytical procedures for determination of trace metals in soil or sediment samples

spectroscopy is used for selenium analysis. Analysis for metals in soil and sediments will be performed by atomic absorption spectroscopy.

Analyses for fluoride are conducted using the ion selective electrode method described by the U.S. EPA.[1] Analyses for nitrate are conducted using the automated cadmium-reduction method described by the U.S. EPA.[2]

Data reporting will consist of sample data and the associated quality control data to document the quality of the analyses. The format of data reports will be mutually agreed to by the prime contractor and ERCO.

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[1] U.S. EPA, 1979, Method 340.2.

[2] U.S. EPA, 1979, Method 353.2.

3.0 QUALITY ASSURANCE OBJECTIVES

The ultimate objective of any laboratory Quality Assurance program is to detect and correct all errors or analytical problems as they occur and guarantee that all data are as accurate and precise as technically possible.

A Quality Assurance program has been specifically designed to meet the requirements of this program. Design of the protocols is the responsibility of Dr. John P. Maney and the system is implemented by the laboratory manager, Mr. Keith A. Hausknecht. In addition, internal chain-of-custody forms have been generated to document the possession of samples in the laboratory. A systematic use of laboratory check standards, interference check standards, standard reference materials, spiked samples, duplicates, and blanks will ensure early detection and identification of any analytical problems that may result. Documentation of quality control data will assist in the detection of any longer-term shifts in the quality of the analyses being performed.

To preclude any transcription or other errors from being included in a final data report, all data will be checked by the laboratory manager, Mr. Keith A. Hausknecht, and the divisional Quality Assurance officer, Dr. John P. Maney, prior to delivery to GCA Corporation.

4.0 DOCUMENT CONTROL PROCEDURES

The purpose of document control is to ensure that all project documents will be accounted for when the project is complete. The prinicpal documents which are the responsibility of the analytical subcontractor, ERCO, are written communications, photographs or drawings, analytical sample tags, laboratory notebooks, and analytical data sheets. ERCO's document control officer for this project is Mr. Keith A. Hausknecht, who is ERCO's Trace Metals Laboratory manager and project manager for the Love Canal Study. The assistant document control officer is Mr. Nile A. Luedtke. These persons will ensure that all documentation are complete and available to GCA Corporation at the completion of the project. All documents will be coded with the appropriate subcontract, laboratory, and document codes as outlined in Section 4.0 of GCA Corporation's Quality Assurance Plan.

5.0 CHAIN OF CUSTODY

ERCO recognizes that proper chain of custody is the most important component of producing court-defensible data. On many projects ERCO utilizes the chain-of-custody procedures which are used by the U.S. EPA/NEIC. The specific components of programs that ERCO commonly uses are as follows:

- When transferring the possession of samples, the transferee signs, dates, and times the reverse side of the chain-of-custody record tag or record. Custody transfers, if made to a sample custodian in the field, are made for individual samples. The chain-of-custody tag or card must be dated and signed by the second person who takes custody. If a third person takes custody, he must follow the same procedure. An additional chain-of-custody tag or card is completed by persons who thereafter take custody. It is apparent, from this chain, that the number of custodians should be minimal. Additional tags or cards should be numbered consecutively.
- If a field custodian has not been assigned, the field sampler is usually responsible for properly packaging and dispatching samples to the proper laboratory for analysis. In that case, the "Dispatch of Sample" portion of the chain-of-custody record tag or card should be properly filled out, dated, and signed.
- All samples are properly packed in shipping containers (e.g., ice chests) to avoid breakage. Ensure that shipping containers are padlocked for shipment to the receiving laboratory.
- A "Sample Transmittal Sheet" is included with all packages. The original and one copy generally accompany the shipment. Copies are mailed directly to the laboratory, to data management personnel, and to any other responsible agent. The survey coordinator retains one copy.
- If the package is sent by mail, it is registered with return receipt requested. If package is hand-delivered, delivery is recorded in the logbook. Receipts are sent from post offices and bills of lading to the laboratory custodians for retention as part of the permanent chain-of-custody documentation.

- When samples are delivered to the laboratory and appropriate personnel are not there to receive them, samples are to be locked in a secure, tamper-proof area. The same person must unlock the samples and deliver custody to the appropriate custodian.
- Internal chain-of-custody is maintained by the Document Control Officer who logs samples in and out on an internal chain-of-custody record (Figure 4).

ERCO has extensive experience with chain-of-custody procedures and will ensure that procedures and documentation are sufficient to meet requirements of GCA Corporation and the U.S. EPA/NEIC.

TRACE METALS LABORATORY

-- CHAIN-OF-CUSTODY RECORD --

ENERGY RESOURCES CO. INC.
185 Alewife Brook Parkway
Cambridge, MA 02138

Sample Identification	Out (Date/Time)	Released By	In (Date/Time)	Received By	Out (Date/Time)	Released By	In (Date/Time)	Received By	Out (Date/Time)	Released By	In (Date/Time)	Received By
Sample No. ____												
Original sample												
Metals digestate: • Hg digestate												
• HNO_3 digestate												
• HNO_3/HCl digestate												
Other (specify)												
Sample No. ____												
Original sample												
Metals digestate: • Hg digestate												
• HNO_3 digestate												
• HNO_3/HCl digestate												
Other (specify)												
Sample No. ____												
Original sample												
Metals digestate: • Hg digestate												
• HNO_3 digestate												
• HNO_3/HCl digestate												
Other (specify)												

Figure 4. Internal chain-of-custody record.

6.0 ANALYTICAL INTERNAL QUALITY CONTROL CHECKS

The internal quality control procedures to be used for this contract are a slight modification of those already employed in ERCO's Trace Metals Laboratory. The following pages are a summary of ERCO's standard quality control procedures as excerpted from the Trace Metals Laboratory Manual.

Project-specific quality control requirements will be implemented if modifications of ERCO's procedures are necessary. These include spikes of the first 10 samples of each type according to analytical procedures provided by GCA. If recoveries do not fall in the required ranges, the method of standard additions will be used. At least one method blank will be processed for every 5-15 samples. One duplicate analysis will be performed for every 10 samples. One laboratory control standard (NBS SRM 1643-a [Trace Elements in Water] or NBS SRM 1645 [River Sediment]) will be run every 5-15 samples to document accuracy of the analytical methods.

QUALITY CONTROL

The laboratory will stand and fall by the quality of work performed. For this reason, a variety of quality control mechanisms have been initiated. The first type of quality control is outlined in the protocols; this includes blanks, handling procedures, etc., which should be followed by all laboratory personnel in performing the analyses. A second type of quality control is afforded by close supervision of the lab managers and lab supervisor. If you have a question regarding any procedure, ASK. A third type of quality control is performed by spot checks in the laboratory. These checks are performed by the laboratory director, division manager and other chemically oriented individuals. A small percentage of errors are, from a practical point of view, impossible to eliminate. The sole purpose of Quality Control and Quality Assurance programs are to identify and correct these errors at the time that they occur.

Quality Control starts with the receipt of chemicals. All chemicals and standards are dated upon arrival. The chemicals with the oldest receiving date are used first. Standards are not used once their expiration date has passed. All standards are kept refrigerated. Standards employed for regulatory work are purchased in duplicate. Two different suppliers (Fisher, Ventron, Spex or MCB) will be used to supply the standards. Before using, these two standards are analyzed and the concentrations compared. If the standards differ in concentration, a third or fourth standard is employed to determine the correct concentration.

A service contract is maintained on all balances. A person cannot make use of a balance unless they have been checked-out by laboratory personnel and their name added to the list of authorized persons posted near the balance. A set of "S" weights will be kept in the Trace Metal Laboratory for the purpose of checking accuracy.

The atomic absorption and inductively coupled plasma (ICP) instrumentation, the water purification system, and clean benches are maintained according to specifications of the manufacturers and appropriate logs are maintained.

All samples for trace metal analysis are acidified or checked for acid preservation upon arrival.

To maintain quality control of results the following methods are employed for all priority pollutant work performed by atomic absorption spectroscopy (AAS).

Minimum Daily Requirements for AAS

- Calibration curves are composed of a minimum of a blank and three standards. A calibration curve is made for every hour of continuous sample analysis.
- A check standard is run approximately every 15 samples. (For priority pollutant work this standard is at the Maximum Contamination Level).
- One duplicate sample is run every 15 samples. A duplicate sample is a sample brought through the whole sample preparation process.
- A minimum of one spiked sample is run every 15 samples. The spiked concentration should be at least 10 times the detection limit concentration.

- Standard deviation is documented for all measurements being made.

- Reported concentrations cannot be less than the lowest standard analyzed.

- Samples are diluted if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

- All preparations employ a minimum of one blank per sample batch.

- If a significant dilution factor would be necessary to analyze a sample by the graphite furnace, the flame atomizer is used.

- Each sample batch requires a minimum of one precision determination and accuracy determination. Accuracy is determined by percent recovery of a spike.

- When available appropriate Standard Reference Materials are analyzed to evaluate new procedures.

- The presence of matrix effects are determined by recoveries of standard spikes to the samples. If recovery is within 10 percent of the expected value, no interference is assumed. This test is performed for every new sample type.

Quarterly Requirements for AAS

- A known reference sample is analyzed once per quarter for the metals measured.

Annual Requirements for AAS

- Unknown performance samples (when available) are analyzed at least once per year.

General Requirements for AAS

- All Quality Control Data are maintained and available for easy reference or inspection using a quality control reference form (Figure 5).
- ERCO's Trace Metals Laboratory routinely participates in the performance of evaluation tests required by U.S. EPA and other federal or state agencies.

Minimum Daily Requirements for ICP

- An instrumental warm-up period of 30 min is required before calibration.
- The average intensities of at least two burns for both the calibration blank and the mixed standards are employed for calibration.
- A check standard is analyzed once every 10 samples. The check standard results must be within 5% of the true values.
- The calibration blank is analyzed once every 10 samples. The results should be within two standard deviations of the mean.
- An inter-element interference check sample is analyzed at the initiation, end, and at periodic intervals throughout the sample run.
- One of every 10 samples will be prepared and analyzed in duplicate.
- A minimum of one sample per every 20 samples is spiked with a multi-element standard and analyzed. If recoveries are not in the 90-110% range, the samples are analyzed by the method of standard additions.
- A procedural blank is required once for every 15 samples or every sample batch, whichever is smaller.

QUALITY CONTROL REPORT FORM

ELEMENT________________

Date________________________

Analyst____________________

ERCO ID#___________________

- SRM_____or CHECK STD._____

  Certified or Prepared conc.________________

  Average Reported Conc._____________________

  % Difference_______________

- DUPLICATES

  Concentration A_____________

  Concentration B_____________

  $$\frac{A-B}{(A+B/2)} \times 100 = \_\_\_\_\_\_\_\_\_\_ \%$$

- RECOVERY

  $$\frac{C \text{ spiked } (\_\_\_\_\_) - C \text{ unspiked } (\_\_\_\_\_)}{C \text{ True Spiked } (\_\_\_\_\_\_\_\_)} \times 100 = \_\_\_\_\_\_\_\_\_\_\_\_ \%$$

Method of Standard Additions Employed? yes_____ no_____

Highest Std run______________ Flame_____ Flameless______

Lowest Std run_______________ N_2O/C_2H_2____________________

Detection Limit______________ Air/C_2H_2____________________

Blank levels_________________ BKG. Corr. yes_____ no_____

COMMENTS:

Figure 5. Quality Control Report Form

- A new sample matrix is evaluated by analyzing serial dilutions and spike additions. The serial dilutions must agree to within 5% and the standard addition recovery must be within 10%.

- Spectral interferences are evaluated using a wavelength scan of the spectral region of interest or comparative testing by atomic absorption spectroscopy should be performed.

Weekly Requirements for ICP

- Calibration standards are compared to a quality control standard, supplied by an outside source, at a minimum of once a week.

General Requirements for ICP

- All quality control data are maintained and available for easy reference or inspection using the appropriate forms (Figures 6 and 7).

- An attempt will be made to participate in performance evaluation and round-robin tests.

Results of quality control determinations by both ICP and atomic absorption are maintained on quality assurance charts (Figures 8 and 9).

ICP SPIKE RECOVERY

ERCO #__________ OPERATOR__________ BG CORR.__________

DATE__________ NEBULIZER__________

Element	Sample Conc.	Spiked Sample Conc.	Difference	Spiked Conc.	% Recovery
Ag					
Al					
Au					
B					
Ba					
Be					
Bi					
Ca					
Cd					
Co					
Cr					
Cu					
Fe					
Fe					
Ga					
K					
Li					
Mg					
Mn					
Mo					
Na					
Ni					
P					

Figure 6. Quality control forms for ICP analysis.

ICP SPIKE RECOVERY (Cont.)

ERCO #__________

Element	Sample Conc.	Spiked Sample Conc.	Difference	Spiked Conc.	% Recovery
Pb					
Pd					
Rh					
Sb					
Se					
Si					
Sn					
Sn					
Sr					
Ta					
Ti					
Tl					
V					
W					
Zn					
Zr					
N+					

Figure 6. Continued.

ICP DUPLICATE ANALYSIS

ERCO #____________ OPERATOR____________ BG CORR.____________

DATE____________ NEBULIZER____________

Element	Conc. I	Conc. II	Average	Difference	% Difference
Ag					
Al					
Au					
B					
Ba					
Be					
Bi					
Ca					
Cd					
Co					
Cr					
Cu					
Fe					
Fe					
Ga					
K					
Li					
Mg					
Mn					
Mo					
Na					
Ni					
P					

Figure 7. Quality control for ICP analysis.

ICP DUPLICATE ANALYSIS (Cont.)

ERCO #____________

Element	Conc. I	Conc. II	Average	Difference	% Difference
Pb					
Pd					
Rh					
Sb					
Se					
Si					
Sn					
Sn					
Sr					
Ta					
Ti					
Tl					
V					
W					
Zn					
Zr					
N+					

Figure 7. Continued.

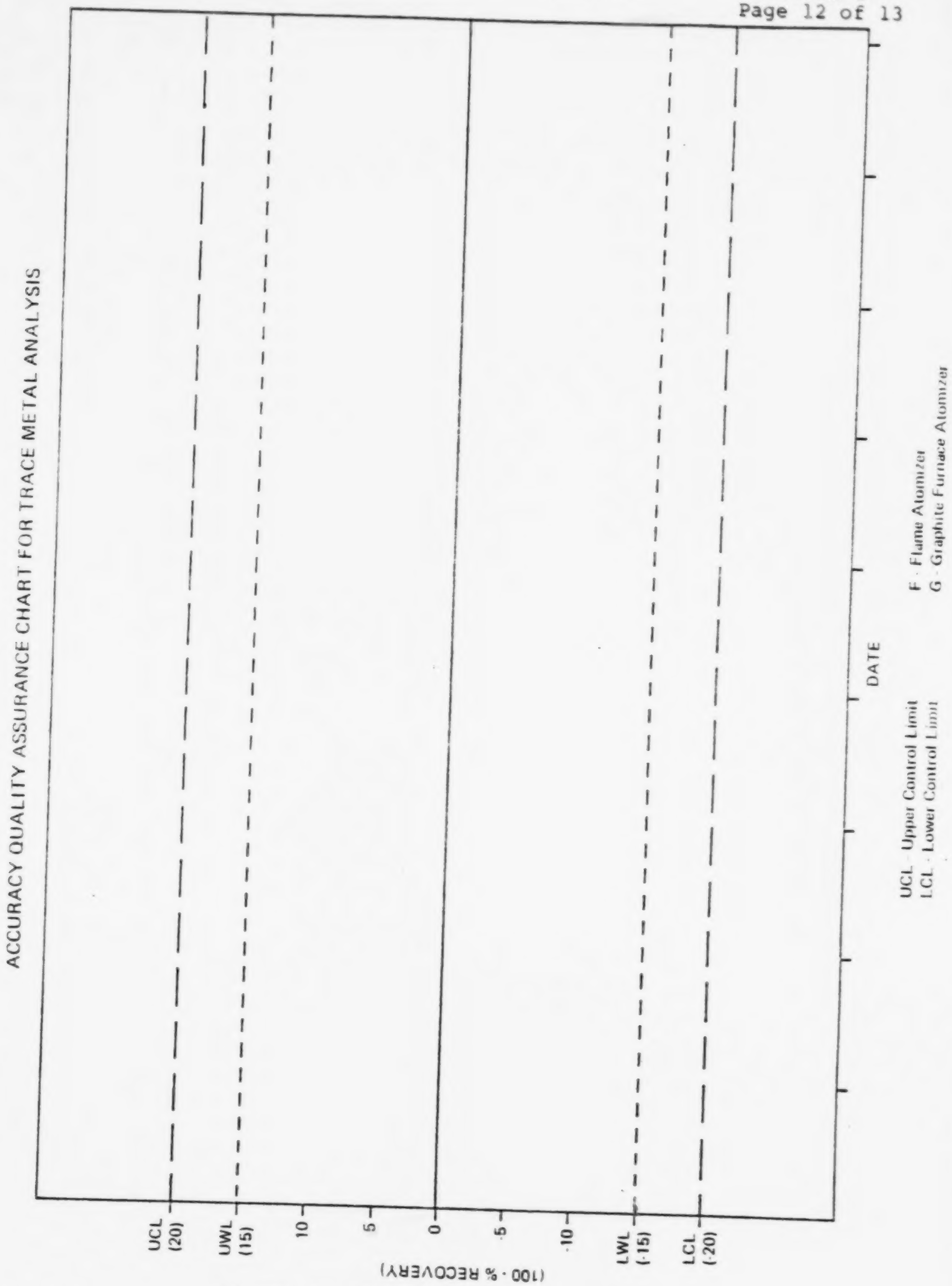
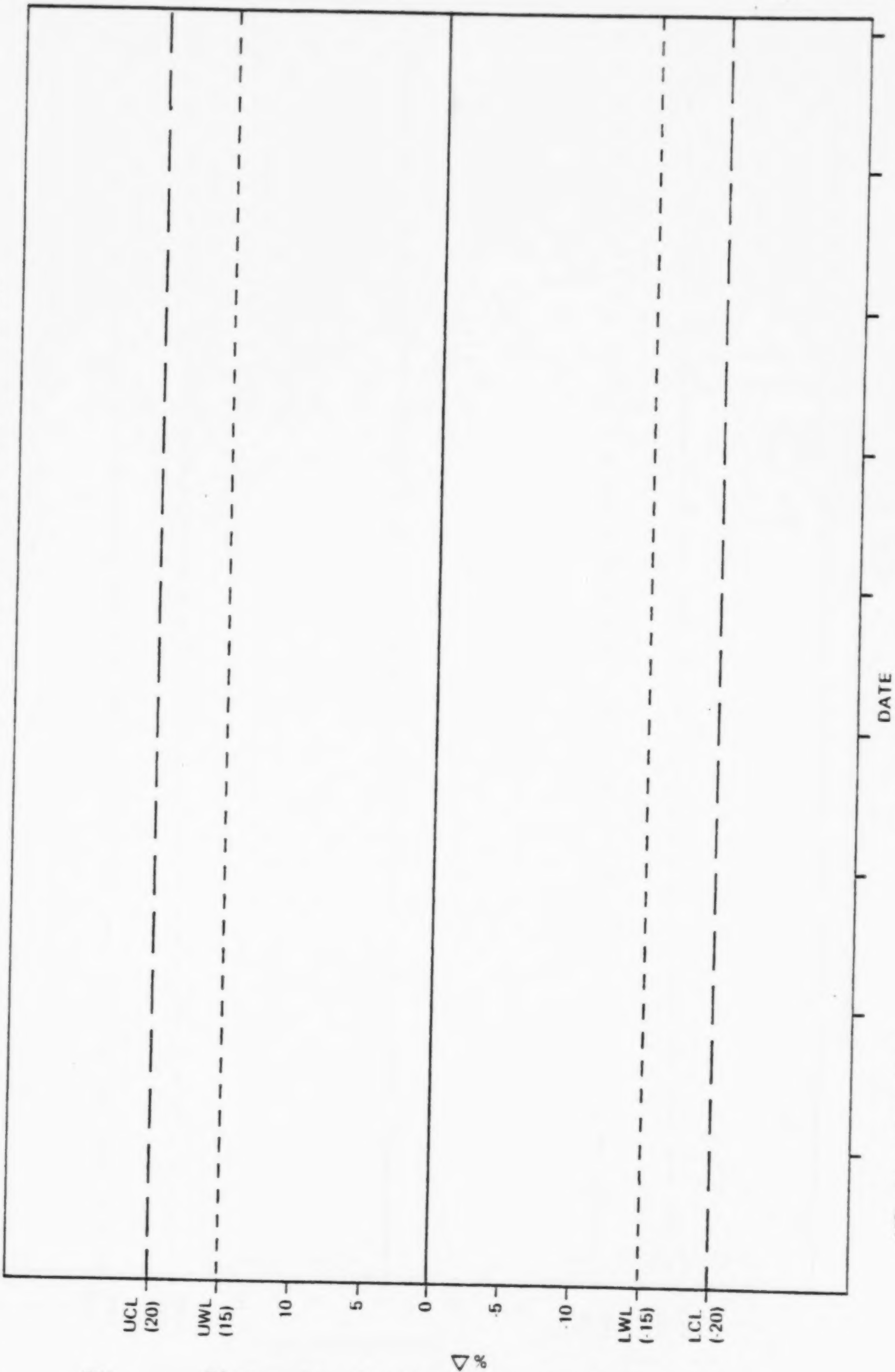


Figure 8. Quality Assurance Chart

PRECISISON QUALITY ASSURANCE CHART FOR TRACE METAL ANALYSIS



UCL · Upper Control Limit
UWL · Upper Warning Limit
LCL · Lower Control Limit
LWL · Lower Warning Limit

C · Concentrations Less Than 15XDL
DL · Concentrations Below Detection Limit
T · Flame Atomizer
G · Graphite Furnace Atomizer

I — In Control (For C Data)
O — Out of Control (For C Data)

Figure 9. Quality Assurance Chart

7.0 DATA REPORTING MANAGEMENT SYSTEM

All data generated during this program will be reported to GCA Corporation using coding forms provided by the prime contractor. Analytical and quality control data will be entered on forms by staff of the Trace Metals Laboratory. Data will be verified prior to transcription by Mr. Keith A. Hausknecht and Dr. John P. Maney to ensure accuracy and adherence to contract requirements. Following transcription, data will be verified for accuracy by Mr. Hausknecht and Dr. Maney before delivery to GCA Corporation. All project documentation will be maintained on-file at ERCO and will be provided to GCA Corporation on request.

8.0 CORRECTIVE ACTION PROCEDURES

Samples will be prepared and analyzed in batches. Each batch will have the appropriate number of duplicates, blanks, spiked samples, and standard reference materials. The analytical results for these quality control samples will be indicative of the calibre of the batch analysis and will be helpful in locating any problems that may occur.

Reagent and procedural blanks will be used to detect contamination from reagents, ambient dust fallout, or unclean glassware. Standard reference materials will be useful in evaluating the efficiency of sample preparation while check standards and spiked and duplicate samples will document the accuracy and precision of the analysis and the analytical program in its entirety.

This system of quality control samples is used to determine if problems are preparation-, standard-, or instrument-related. After identifying the source of the problem, corrective measures are then a matter of routine.

Implementation of quality control in the laboratory is the responsibility of Mr. Hausknecht. He monitors the progrss and quality of work on a daily basis and is notified by staff members if results do not meet project requirements. Analytical procedures are then examined in consultation with Dr. Maney to determine the source of the problem and corrective action is taken. These actions may include, but are not limited to, preparing new standards or reagents, recalibrating the analytical instruments, repairing the analytical instruments, preparing new samples, reanalyzing sample digestates, and analyzing samples by standard additions. Quality assurance is monitored by Dr. Maney on a weekly basis. If quality assurance protocols do not meet project specifications, the laboratory manager is notified and procedures are reevaluated to ensure adherence to contract requirements.

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APPENDIX B

ANALYTICAL EQUIPMENT OPERATION, MAINTENANCE, AND TROUBLE SHOOTING PROCEDURES

FLAME ATOMIC ABSORPTION

The instrument should be operated using the manufacturer's suggested settings. A 15-20 minute warmup of hollow cathodes should be sufficient for a double-beam system. However, more time may be required for certain elements such as As and Se when using background corrections, since the Perkin Elmer 603 is single-beam in the background correction mode. The manufacturers' listed sensitivity should be met or surpassed. If a lack of sensitivity is found, burner position and flame conditions should be optimized while aspirating a high standard. If this doesn't suffice, a new standard should be made and optimization performed a second time. If unsuccessful, one should note the flame conditions, looking for the presence of acetone (yellow-orange flicker). A very bad tank of acetylene will also leave an oily residue around the burner head slot. If the flame appears to be normal, then the nebulizer's uptake rate should be checked. If the uptake rate is not within specifications then the nebulizer should be readjusted. If the uptake rate is within specification, the sensitivity of a second element should be checked to see if the problem is element dependent. At this time the laboratory supervisor should be notified.

When sample size allows, a minimum 5-second integration period should be employed to minimize noise.

Samples should be free of particulate matter and be similar to the blank in terms of viscosity.

The lower noise levels encountered when using a secondary wavelength for high concentration analyses make it the preferred method over rotation of the burner head.

Ionization suppressants should be added when analyzing alkali metals in air/acetylene flames or alkaline earth metals in nitrous-oxide/acetylene flames.

The analysis of polyvalent metals in the presence of polyvalent anions requires the use of a releasing agent such as lanthanum or a nitrous-oxide/acetylene flame to preclude chemical interference.

When background correction is employed, the alignment of the deuterium arc beam with the hollow cathode beam should be checked as described in the instruction manual (Section 8-28, Para. 12). Background correction must be used when analyzing any element whose resonance line has a wavelength shorter than 3000 Angstroms. Background correction should be used whenever the deuterium arc intensity can be matched to that of the hollow cathode and not result in a serious decrease in the signal/noise.

ANALYTICAL TROUBLESHOOTING FOR FLAME ATOMIC ABSORPTION

PROBLEM	POSSIBLE CAUSE	CORRECTIVE ACTION
Excessive noise with low energy and flame is extinguished	Low hollow cathode current	Use manufacturer's current setting.
	Incorrect bandpass	Use manufacturer's suggested slit setting.
	Incorrect spectral line	Scan monochrometer for a more stable resonance line. Check observed sensitivity against manufacturer's suggested sensitivity.
	Dirty optics	Clean optical winddows in hollow cathode and burner component.
	Other	Consult P.E. Manual, ¶ 11.H.1.
Excessive noise which occurs with flame	Incorrect burner alignment	Ensure that optical path is not passing through edge of flame or too high or too low in flame.
	Improperly adjusted nebulizer	Adjust according to ¶ 8.C.8 of P.E. Manual.
	Precipitate in sample or standards	Make new standards. Allow particles to settle to bottom of sample container or digest.
	Other	Consult P.E. Manual, ¶ 11.H.2.

ANALYTICAL TROUBLESHOOTING FOR FLAME ATOMIC ABSORPTION (CONT.)

PROBLEM	POSSIBLE CAUSE	CORRECTIVE ACTION
Low Sensitivity	Incorrect standard or too old or no ionization supressant	Make a new standard.
	Incorrect flame conditions	Consult methods manual. Be cautious with elements that require reducing flame.
	Incorrect burner adjustment	Ensure that optical path is not passing through the edge of the flame or too high or too low in flame.
	Incorrect bandpass	Use manufacturer's suggested slit setting.
	Incorrect spectral line	Scan monochromatic for a more sensitive and correct line.
	Hollow cathode current too high	Use manufacturer's suggested current setting.
	Poor grade of acetylene	This problem will be especially noticeable when analyzing for AL, Si, and CA. Consult maintenance section of this manual.
	Other	Consult P.E. Manual ¶ 11.G.4.
Hollow cathode lamp will not light	BKGD CORR switch is set on BG	Reposition switch to AA-BG.
	Defective lamp	Check other lamps to determine if problem is with the lamp or the instrument.

ANALYTICAL TROUBLESHOOTING FOR FLAME ATOMIC ABSORPTION (CONT.)

PROBLEM	POSSIBLE CAUSE	CORRECTIVE ACTION
Electrodeless discharge lamp will not light.	No source of seed ions	Use an ultraviolet light to ionize the Ar fill gas and seed the lamp with seed ions.
Error code "C" appears on the digital display	Various reasons	Consult P.E. Manual ¶s 11.C.1-4; 11.E.3,4,6; 11.F.3.
Digital display malfunctions.	Various reasons	Consult P.E. Manual ¶ 11.D.
Malfunction in the concentration mode.	Various reasons	Consult P.E. Manual ¶ 11.F.
Results erroneously high.	No blank correction standard Bad standards Background absorption	Consult P.E. Manual ¶ 11.G.1.
Results erroneously low.	Chemical or physical interference Bad standards Contaminated blank	Consult P.E. Manual ¶ 11.G.2.
Poor detection limits	Scale expansion settings Integration time settings Incorrect lamp settings Improper flame conditions Poor sensitivity	Consult other subsections in this section. Consult P.E. Manual ¶ 11.G.3.

ANALYTICAL TROUBLESHOOTING FOR FLAME ATOMIC ABSORPTION (CONT.)

PROBLEM	POSSIBLE CAUSE	CORRECTIVE ACTION
Baseline drift in background correction mode	Single beam functioning of instrument in the background correction mode	Balance hollow cathode and deuterium arc intensities and re-zero.
Baseline drift when not in background correction mode	Burner warm-up Blocked capillary Lamp positioning	Consult P.E. Manual ¶ 11.I.1.
Gases do not ignite.	Gases off Incorrect gas setting Safety cable not inserted	Consult P.E. Manual ¶ 11.K.1.
Flash back	Incorrect gas settings Incorrect gas connections Leaks	Consult P.E. Manual]11.K.2.

FLAMELESS ATOMIC ABSORPTION

The instrument should be operated using the manufacturer's suggested settings. A 15-20 minute warmup of hollow cathodes should be sufficient for a double-beam system. However more time may be required for certain elements such as As and Se when using background correction, since the Perkin-Elmer 603 is single beam in the background correction mode. Electrodeless Discharge Lamps (EDLs) may require a longer time period to minimize noise levels.

Alignment of the furnace should be carried out in the manner described in the HGA-2100 instruction manual.

The initial temperature settings for the dry, ash, and atomization mode should be as the manufacturer suggests. If, however, a significant background is encountered during atomization a higher ashing temperature may be possible and may improve precision. The maximum ashing temperature is determined by rerunning the sample in succession while monitoring the atomization peak height. When the peak height starts to decrease, the maximum ashing temperature has been surpassed. The ashing temperature at this time should be returned to the previous setting.

Background absorption is due to molecular absorption and scattering by particles as matrix materials cool in the graphite tube during atomization. Samples with high acid content, or high dissolved solids concentrations, can experience significant background when analyzing lower wavelength elements. For these samples the "gas interrupt mode" should not be employed since it will increase the residence time of these compounds in the optical path offsetting any advantages.

The atomization temperature should be sufficiently high so as to cause complete atomization of the element of interest.

The optimum atomization temperature can be determined by a sample run followed by a blank run with no high temperature cleanup between runs. The blank run should not display any absorbance, if the atomization temperature was high enough. If an absorbance did occur then the atomization temperature should be raised. The atomization temperature should not be set excessively higher than required since it will cause unnecessary wear of the graphite components.

If background is employed, the alignment of the deuterium arc beam with that of the hollow cathode should be checked as described in the in the 603 instruction manual (Section 8-28, Para. 12). Background correction must be used when analyzing any element whose resonance line has a wavelength shorter than 3000 Angstroms. Background correction should be used whenever the deuterium arc intensity can be matched to that of the hollow cathode and not result in a serious decrease in the signal/noise.

Analytical Trouble Shooting for Flameless Atomic Absorption

Problem	Possible Cause	Corrective Action
Low Sensitivity	Wrong wavelength.	Reset monochromator.
	Slit size too large.	Use slit setting recommended in Methods Manual.
	Incorrect Standard.	Make up fresh standards.
	Dry cycle too short.	Be sure sample dries completely before ash cycle.
	Ash cycle too hot.	Lower ash temperature to prevent volitilization of the element being analyzed during ash cycle.
	Atomize temperature too low.	Raise atomize temperature to allow element being analyzed to vaporize rapidly and not just creep up to a peak.
	Sample volume too small.	Check that correct pipette is being used.
	Instrument is overdamped.	Use lowest possible damping to the recorder.
	Argon flow is too high.	Lower argon flow to approximately 2.5 liters/hours (for most elements with the new style head).
	Graphite tube is worn.	Replace graphite tube.
	Ramping control is on.	Set ramping control to zero unless its use is required.
	Atomize head incorrectly aligned.	Align atomizer head for maximum light transmission through the graphite tube.
	Atomizer head is too hot for pipetting sample.	Check for a steady water flow through the head. Wait longer between samples.

Problem	Possible Cause	Corrective Action
Poor Reproducibility	Atomizer head too hot when pipetting samples.	Check water flow. Wait longer between sample pipetting.
	Argon flow too high.	Lower argon flow to approximately 2.5 liters per hour on new head.
	Poor precision of sample pipetting.	Check pipet precision. Do not allow pipet to touch sides of graphite when inserting the tip.
	Poor contact between atomizer head and graphite tube.	Clean points of contact.
	Some sample lost during ash cycle.	Lower ash temperature to prevent volitilization of the element being analyzed.
	Sample splatter during dry cycle.	Lower dry temperature to prevent splattering. Increase drying time to allow complete drying of sample before ashing.
	Graphite tube is porous.	Replace graphite tube.
	Too large a sample.	Do not exceed 50 µl sample size.
	Sample not completely atomized.	Increase atomization temperature and/or increase atomization time.
	Build-up of sample.	Use clean-up operation after each sample.
	Background absorption from sample matrix.	Use higher ash temperature if possible. Use ramp mode if applicable.

Problem	Possible Cause	Corrective Action
		Use simultaneous background correction.
	Sample not homogeneous.	Good sample dissolution procedure necessary.
	Sample may be forming a carbide complex with the graphite tube.	Avoid high concentrations of this element. Sample analysis may not be practical with graphite tube.
Graphite Tube "Memory"	Too large a sample being used causing sample to flow to cool ends of tube.	Do not exceed 50 μg sample size. Do not saturate tube with samples of high concentrations.
	Previous sample may not have been completely atomized.	Increase atomization temperature and/or time. Use clean-up operation between samples.
	Sample may be forming a carbide compound and not completely atomizing.	Increase atomizer temperature or time. Use clean-up operation between samples. Sample may not be practical for graphite tube analysis.
	Graphite tube may be contaminated.	Replace graphite tube if repeated clean-up attempts do not solve the problem.
	"Memory" may be due to the intense radiation of graphite tube saturating the photomultiplier tube.	Increase hollow cathode lamp current up to maximum rating if necessary. Use smaller slit size in conjunction with increased hollow cathode current.

Problem	Possible Cause	Corrective Action
Short Graphite Tube Life	Argon flow too low.	Argon flow should be at least 2.5 liters per hour. For some elements requiring a very high atmoization temperature increase the flow to 4.0 liters per hour flow.
	Air-ash temperature too high.	High air-ash temperatures will decrease tube life and should be avoided if possible.
	Tube clean-up operation while in Auto-2 cycle.	Argon flow must be on during tube clean-up cycle. Switch to continuous flow for clean up operation.
	Excessive atomize temperature.	Lower atomize temperature if possible to prevent graphite tube deterioration.
	Graphite tube installed improperly.	Check instruction manual for tube installation.

INDUCTIVELY COUPLED PLASMA

The following passages have been selected from the manufacturer's user's manual which is used as a guide for the routine operation and troubleshooting of the ICP spectrometers.

The inductively coupled plasma is a precision electronic instrument designed for trace metal determinations and should therefore be used in an area separated from wet benches and storage areas of acids, corrosives and volatile materials as these vapors may attack the surfaces of the optical components and electrical wiring. Similarly, while it is not essential to operate the instrument in a "clean room," the presence of dust in the laboratory atmosphere will degrade the analytical performance of the instrument at ultra-trace levels. Additionally, it is essential that the analytical sample solutions and standards be free from particulate material such as dust since this can plug the nebulizer and give anomalous results.

The ambient temperature may not exceed 26.6°C (80°F) and the relative humidity may not be in excess of 60 percent.

Operating the Plasma

Always wear safety goggles or UV protective glasses when viewing the plasma directly as the intense ultraviolet radiation produced will damage the eyes.

Initiating the Plasma with Aqueous Solutions

1. Turn on power to the RF generator, but do NOT turn on RF ouput. Turn on the exhaust fan. Turn on the argon - if the gas pressure at the input has been changed it may be necessary to re-adjust the flow controller.

2. Set the coolant gas flow to 20 l/min. Allow the nebulizer to aspirate water for a least three minutes before igniting plasma to purge the torch of air. The gas flows used for this step are not critical.

3. Turn on the water supply at the external laboratory source.

4. When the RF generator has reached standby, the plasma can be initiated. Set the power adjustment dial on the auto power control unit to the run value. All the values for generator operation are tabulated in the operation sheet included with calibration data.

5. Set the coolant flow to 25 l/min and the plasma gas flow to 1 l/min. Turn off the sample gas flow by rotating flow meter control clockwise and be sure that water is no longer being aspirated before continuing.

6. Push the ignitor button once and make sure a discharge occurs through the argon flow. If this does not occur, check that the discharge is not grounding, the coolant gas flow is correct, and the tesla lead is connected.

7. Turn the power control dial on the generator to minimum (fully counter clockwise) and switch on RF power. Set

the capacitor on the side of the plasma housing to the start value. Switch auto power control unit of the generator to manual. Push the ignitor buttion and the rotate power control dial until the plasma ignites.

8. Once the plasma has been initiated, disengage the ignitor button and check that tail flame is not yellow - if it is, switch off immediately as the quartz is overheating. Allow the torch to cool and check the position of the torch and that the plasma gas flow rate is 1 l/min and that the sample gas is NOT flowing. Increase the coolant flow before trying to reignite the plasma.

9. If the plasma is burning correctly, rotate the capacitor control located on the side of the plasma to the predescribed run value (this brings the reflected power below about 50 W). Increase the forward power to about 1.5 kW by rotating the power control dial. Switch the auto power control unit of the generator to automatic and rotate the power control fully clockwise.

10. Flip off the toggle valve below the plasma gas flow and slowly rotate the sample gas flow control counterclockwise until it reads the value listed in the operational sheet (normally about 1 l/min after two complete turns). Verify that solvent is being aspirated. This will be observed as a darker central channel in the plasma discharge.

11. Reduce the coolant flow slowly to the value on the operation sheet (about 15 l/min). This is achieved by rotating the knob on the flow meter counterclockwise.

12. Tune the plasma by rotating the side capacitor so that reflected power is minimized. On the first ignition, when the torch has been reinstalled and or repositioned, quickly open the front door of the plasma housing (this switches off the plasma) and check that the tip of the aspirator is not glowing red, after about 2 minutes of aspirating solvent into discharge. If the tip glows, lower the torch by 1 mm and reignite the plasma.

13. During the first 20 minutes or so, it is necessary to check the match as drift may occur during the instrument warm-up. In general, aqueous type samples should be run at about 1 to 1.25 kW forward power.

14. Using the vertical and horizontal adjustments of the imaging system, focus the discharge on the spectrometer entrance slit such that the work coil image is at the value on operation sheet (about 20 mm) below the entrance slit on the front projection screen (the top of the torch should then be about 12 mm below the slit). Aspirate 1000 ppm Y into the plasma and adjust the sample throughput to the discharge until the tip of the red tongue is at the value on the operation sheet (about 13 mm below the entrance slit - that is about 1 mm below the open end of the torch). Sample throughput to the torch and hence the position of the Y red region is adjusted via the pressure control valve. Clockwise rotation of the pressure control valve will bring the Y red region closer to the slit. The blue Y atom-rich region is then viewed by the spectrometer. The system is then ready for optimization.

Optimization of Plasma Operating Conditions

In general, it is possible to fully optimize the plasma for only a selected group of analytical channels at any one time and this depends upon the spectrochemical properties of the analyte. Thus, Na, an easily ionized element, is normally determined using an atomic line in the visible region of the spectrum, i.e. 589.0 nm, while Cd is normally determined at its ionic lines in the ultraviolet region of the spectrum, i.e. 226.5 nm or 214.4 nm. It is obviously not possible simultaneously to optimize conditions for these two elements since a high temperature is required in order to ionize Cd and produce intense ionic Cd emission which will also cause a high degree of ionization of Na and hence produce a low atomic population. A cooler plasma, on the otherhand, will favor Na atomic emission, but the discharge will then have insufficient energy to ionize Cd to the same extent as a hotter plasma. Thus, it is necessary to operate the plasma at compromise conditions for a wide range of elements.

The element chosen for optimization of the plasma should have an analytical line in the first order of the spectrometer and should be sensitive so that a 1 ppm concentration will give a signal to a blank solvent ratio of a least 2 at optimization. (This means that the offset should be less than 1 ppm, preferably less. Offsets are supplied in the log-book together with the calibration information determined at Jarrell-Ash.)

It is recommended that one of the following lines be used for optimization:

Cd 226.5 or 214.4 nm
Zn 206.2 or 213.8 nm
Co 238.8 or 228.6 nm
Ni 231.6 nm
Mo 202.0 or 203.8

Standardization of the Plasma

Since the inductively coupled argon plasma exhibits such a high degree of linearity, it is necessary only to use a two point standardization for each element to cover the analytical range of the instrument.

In practice it is recommended that for all elements the low standard be the reagent blank - ZERO concentration. The value of the high standard should be such that it lies near or above the top of the normal range in the matrix to be used, provided that this value is at least equal to one-half of the offset or 0.5 ppm, whichever is the higher. In certain cases, where two lines are used to extend the linear range for a particular element, e.g. Ca 393.3 nm and Ca 370.6 nm or Na 589.0 and Na 330.3, then the value of the high standard should be chosen to correspond to the cross-over point between the two lines.

Interference Effects and Inter-Element Corrections

Spectral type interferences do occur with the plasma system and these can be compensated for by use of group three in the matrix program. In order to determine the magnitude these effects, the following steps should be followed:

1. Optimize and standardize the plasma as described in Sections B3 and B4.

2. Run a 1000 mg/1 solution of the interferent element and perform at least two burns on the sample being printed. The interferent element solution must be spectroscopically pure. After the analysis has been performed, aspirate a water blank for several minutes until the interferent element concentration reaches the blank level. Then aspirate the next elemental solution until all elements in the matrix have been analyzed.

After all the data has been determined, it is then necessary to decide which inter-element corrections to apply. The inter-element correction factor is then calculated and input in group three of the matrix program.

Midwest Research Institute
QA Plan

SECTION 1.0

PROJECT DESCRIPTION AND ORGANIZATION

This project involves the analysis of adipose tissue collected from animals at Love Canal. The analytical scheme entails a solvent extraction and the determination of the level and identification of a selected group of compounds in tissue by gas chromatography/mass spectrometry (GC/MS).

The personnel assigned to this project are:

John Hosenfeld, Project Leader, responsible for the overall technical and fiscal goals of the project.

Evelyn Conrad, Quality Control Coordinator, prepares internal QC standards for blind samples.

Paul Cramer, Project Chemist, assists the project leader in the technical aspects of the project.

Ann Small and Kay Turman, Extraction Chemists, responsible for sample preparation and extraction.

Gil Radolovich and Margie Wickham, Analytical Chemists, responsible for analysis of the samples by GC/MS and first order data review.

Hope Miller, Document and Sample Control Officer, responsible for document and sample control.

The organizational structure for this project is included in Figure 1.

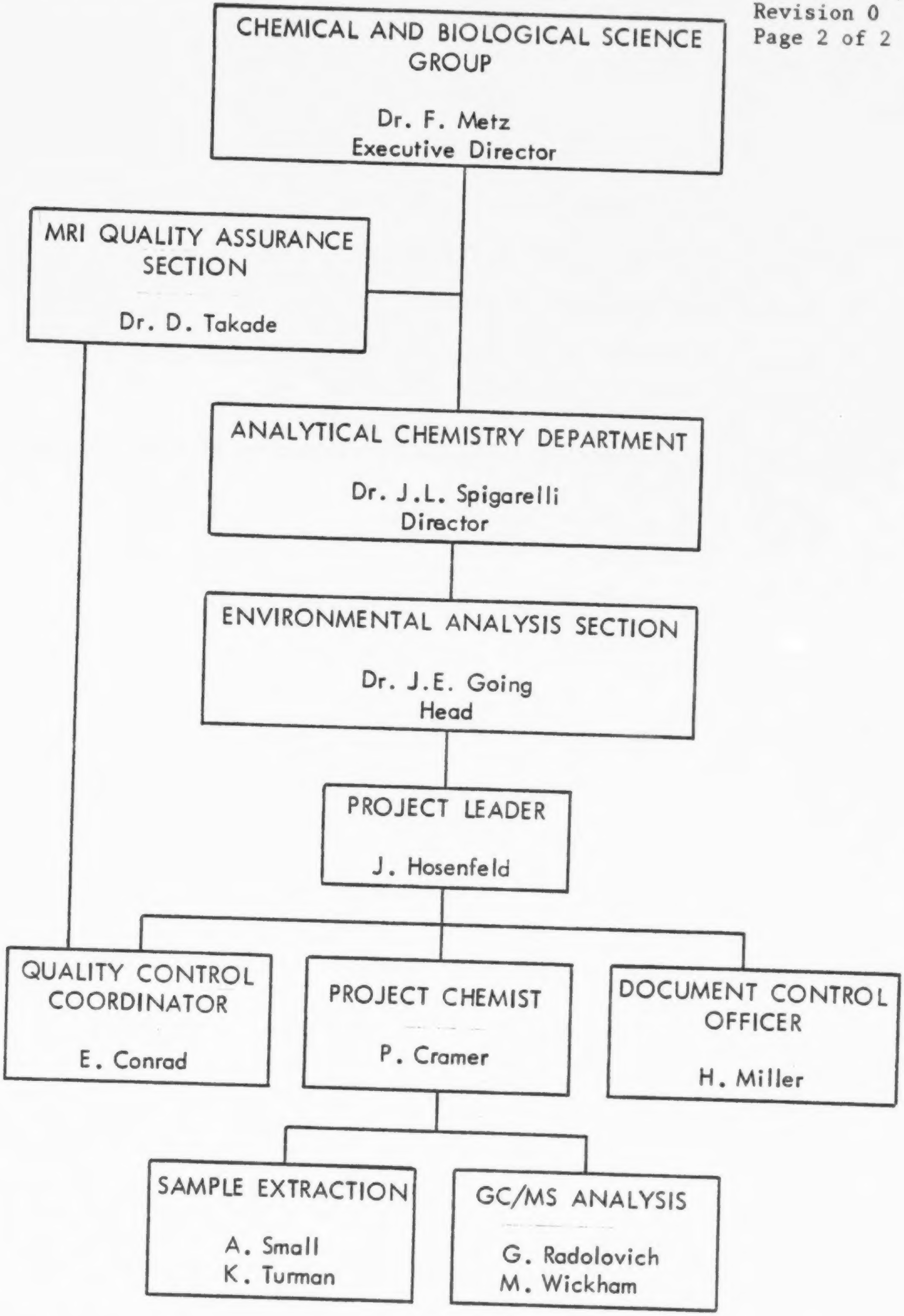

CHEMICAL AND BIOLOGICAL SCIENCE GROUP
Dr. F. Metz
Executive Director
MRI QUALITY ASSURANCE SECTION
Dr. D. Takade
ANALYTICAL CHEMISTRY DEPARTMENT
Dr. J.L. Spigarelli
Director
ENVIRONMENTAL ANALYSIS SECTION
Dr. J.E. Going
Head
PROJECT LEADER
J. Hosenfeld
QUALITY CONTROL COORDINATOR
E. Conrad
PROJECT CHEMIST
P. Cramer
DOCUMENT CONTROL OFFICER
H. Miller
SAMPLE EXTRACTION
A. Small
K. Turman
GC/MS ANALYSIS
G. Radolovich
M. Wickham

Figure 1 - Project Organization

SECTION 2.0

MAJOR PROJECT ELEMENTS

The major project elements are as follows:

- Establish chain of custody procedures
- Receipt and inventory of samples
- Sample extraction and analysis
- Data review and report

Each of these elements is described in subsequent sections of this plan.

SECTION 3.0

QUALITY ASSURANCE OBJECTIVES

The purpose of this plan is to provide data which has a level of precision and accuracy consistent with current state-of-the-art techniques. Adipose tissue has been analyzed for pesticides and a method has been provided to MRI by the prime contractor. However, the combination of the extraction method with the analysis section of the EPA Method 625 has not been validated. Consequently, a firm level of precision and accuracy cannot at this time be stated. However, MRI has experience in the analysis of priority pollutants in fish. In this work spiked samples of fish tissue were analyzed by the GC/MS system. From these data, information on the precision and accuracy of the analytical methods was obtained. These levels showed a precision of ± 20% and an accuracy of ± 15%. We anticipate achieving these levels in the current project.

SECTION 4.0

DOCUMENT CONTROL PROCEDURES

Document control is an integral part of each project at MRI. Bound technical record books are assigned, as required, to each project by the MRI security office. Each record book is assigned a unique number and signed out by the project leader who then maintains control of the book for the duration of the project. The record book contains consecutively numbered pages which are filled out whenever activity occurs on the project. A signature block is provided on each page for the person who performs the work and enters the data. A separate signature block is provided for the signature of the individual to whom the data is explained. At the completion of the project, the record books and all other pertinent project information are stored in the MRI vault.

For this project two technical record books will be used. The first book will be used for sample inventory, and descriptions of sample preparation and handling. The second record book will be used for entry of the analytical procedures and data summaries from the GC/MS. Both books will be maintained by the project leader, or in his absence by the project chemist.

SECTION 5.0

CHAIN OF CUSTODY AND SAMPLE CONTROL

Chain of custody procedures will be enforced during the entire tenure of project. The samples and any subsamples obtained therefrom will be maintained in a locked facility.

The sample container will be transported to a high security area at MRI. The container will then be opened and each sample will be assigned a number unique to this project. Each sample number will be entered into a notebook along with the date of receipt, sample description, type and container (i.e., bottle, ampule).

This procedure will be carried out under the supervision of the project leader. The sample inventory control procedures depend upon an unique sample identification system. The proposed sample coding system will be used to identify the samples which are received for analysis at MRI. The sample will maintain the MRI code throughout the analytical process. All analytical data for the sample will include the MRI sample code.

The coding system to be used will follow the general format of sample number, project designation (LC for Love Canal), and subsample identification. The subsample identification will be used when either replicates or spikes are included in the analysis. For example, 12-LC-2 represents sample number 12 from the Love Canal project and is the second replicate of that sample. An aliquot of sample 10 on the Love Canal project that was the duplicate sample to which the second spike level was added would have the designated I.D. number of 10-LC-2-2. The sample identification number then will become an integral part of the sample transfer mechanism.

In order to establish, maintain and control sample traceability, each sample set will be handled as a unit. These samples will be maintained separately from any other sample sets.

Chemists working with the samples will acknowledge receipt of these samples with their signature on the sample transfer form. Each transfer must then be counter-signed by the sample control officer as shown below.

Sample Transfer Form

Sample Identification Numbers ______________________

These samples (I.D. numbers) are hereby transferred to:

Receiver of Sample ______________________

Custodian of Sample ______________________

at __________ o'clock on ______________________ (date)

The receiver is responsible for maintaining sample control until relieved from this duty in writing. The sample transfer form will be maintained as a single unit by the Document Control Officer. In every instance, each entry into a notebook or other recording system must include as a minimum the following information: sample number, date of entry and any sample manipulations conducted on that day of entry.

SECTION 6.0

ANALYTICAL INTERNAL QC CHECKS

The Quality Control system for this project shall be conducted in two phases. Phase 1 will consist of the analysis of spiked samples of rat adipose tissue obtained from rats in the Animal Housing Facility at MRI. These rats will not have been exposed to any of the analytes of interest prior to obtaining the tissue. The tissue will be spiked with 1,4-dichlorobenzene, p,p'-DDE and chrysene at two levels. These levels will be approximately 20 ppm and 2 ppm.

The Quality Control samples for Phase 2 will consist of MRI Priority Pollutant standards which will be adjusted to known concentrations by the QC coordinator and submitted as blind samples to the GC/MS center. Additional blind QC samples will be randomly chosen from the list supplied by the prime contractor. All QC blind samples will be included with the actual samples which are sent to the GC/MS center for analysis. Approximately 20% of the total number of samples analyzed will be QC samples.

Control charts will be prepared for the three compounds which are used to spike the tissue samples. These charts will be used to monitor the day to day variability of the analytical procedures and more importantly will provide a means to initiate corrective action if the results fall outside the established control limits of two standard deviations from the mean. Examples of previous control charts used for GC/MS analysis are included in Figures 2 and 3.

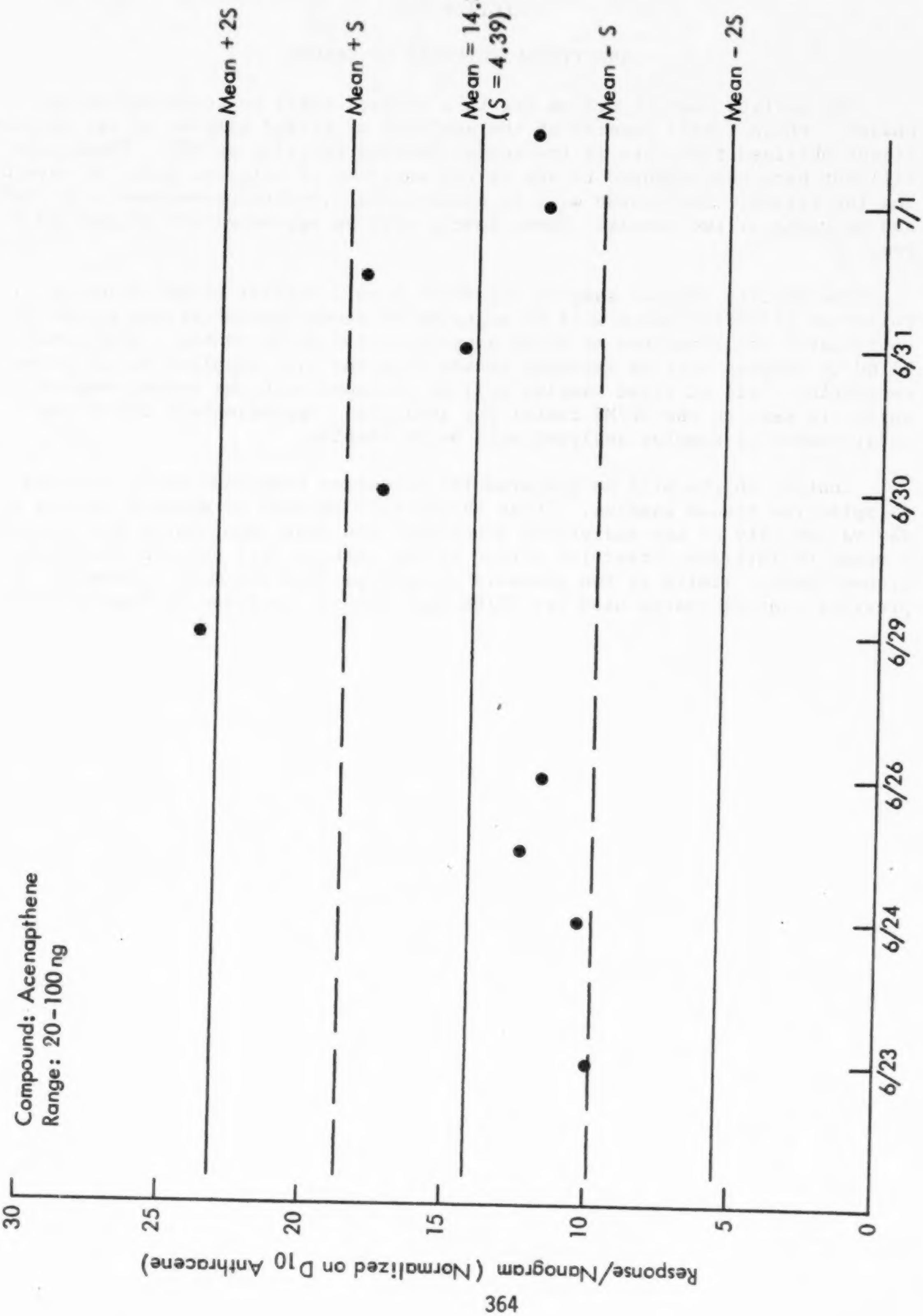


Figure 2 - Control Chart for Acenapthene

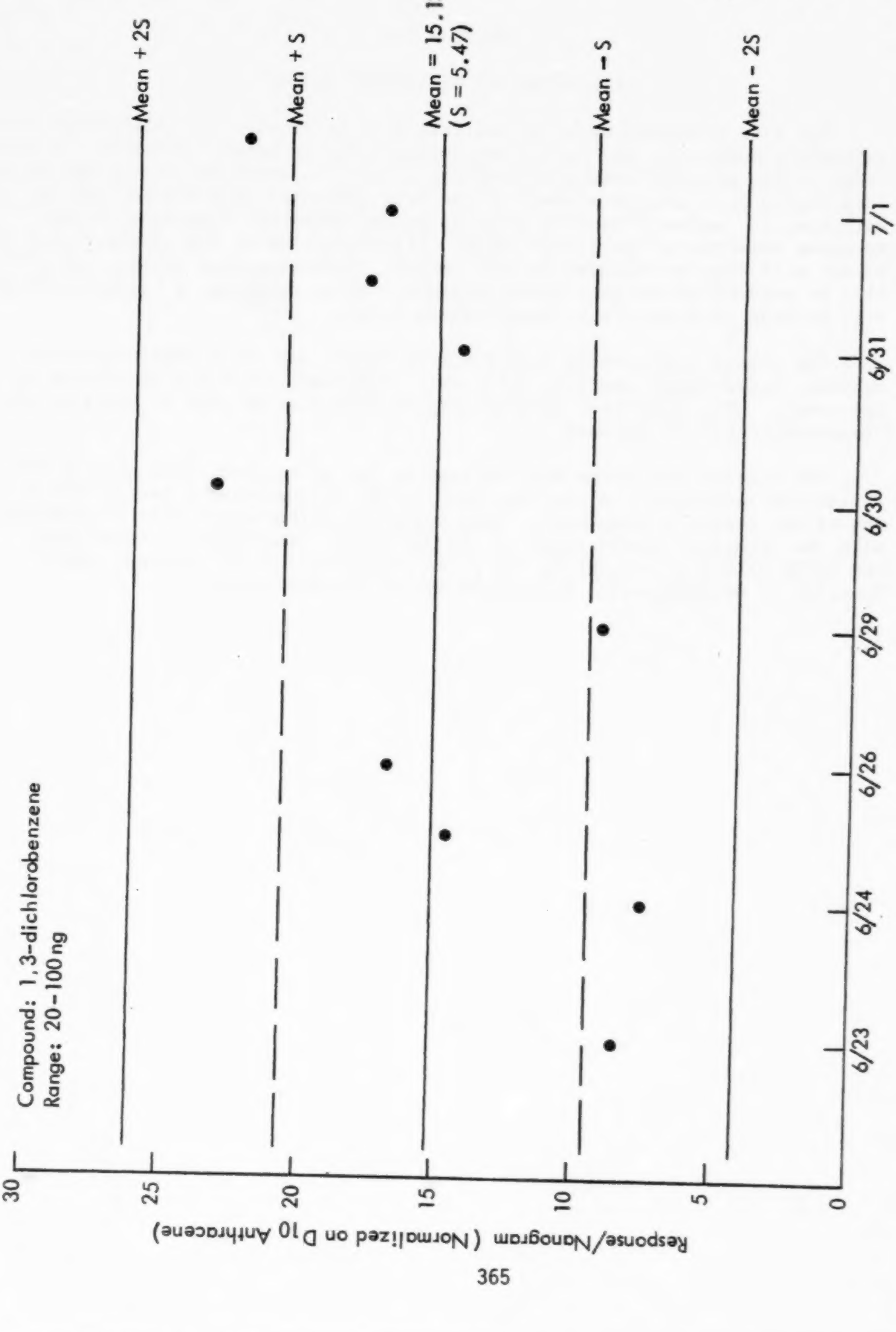


Figure 3 - Control Chart for 1,3-Dichlorobenzene

SECTION 7.0

DATA REPORTING MANAGEMENT SYSTEM

The data generated by GC/MS analysis will be encoded with the sample identification numbers on the data forms supplied by the prime contractor. A first order review of these data will take place prior to entry of data on the forms. This review will involve a check of the data generated by the GC/MS system with specific emphasis on such items as proper retention times and analyte response relative to the linear range. The inspection of the spiked recovery values will also be included in this review. Following this review, the data will be entered on the data forms provided. After encoding, a clerical check will be made to correct any transcription errors.

The second order review will focus on inter- and intra-sample analysis results. Intra-sample analysis will review compounds which are identified in the sample. The comparison between samples will also be made to check on the "reasonableness" of the data.

The encoded data forms will be sent to the prime contractor and the EPA designated laboratory. A copy retained at MRI will provide a backup should any of the copies be misplaced. Hard copies of GC/MS output will be archived with the technical record books in the MRI vault. The magnetic tapes with the GC/MS data will be sent to the prime contractor by air courier. The transfer of documents will be managed by the project leader.

SECTION 8.0

CORRECTIVE ACTION PROCEDURES

The implementation of corrective action in the analytical procedures will be effected in a two level approach. The first level involves the GC/MS and its response to a known standard. A generalized diagram of the decision/action process involved in the GC/MS analysis is included in Figure 4. This corrective action would resolve problems associated with the operation of the GC/MS system. However, if a problem is evident from the recovery spikes, replicates or blanks, then the second level of corrections will be undertaken. The identification of second level problems will be the responsibility of the chemist and project leader. The appropriate corrective actions will be indicated by review of analytical data for method blanks and spiked blanks to ensure that the proper sample preparation procedures had been followed. Additional cleanup procedures for the florisil, sodium sulfate or the glassware may then be required.

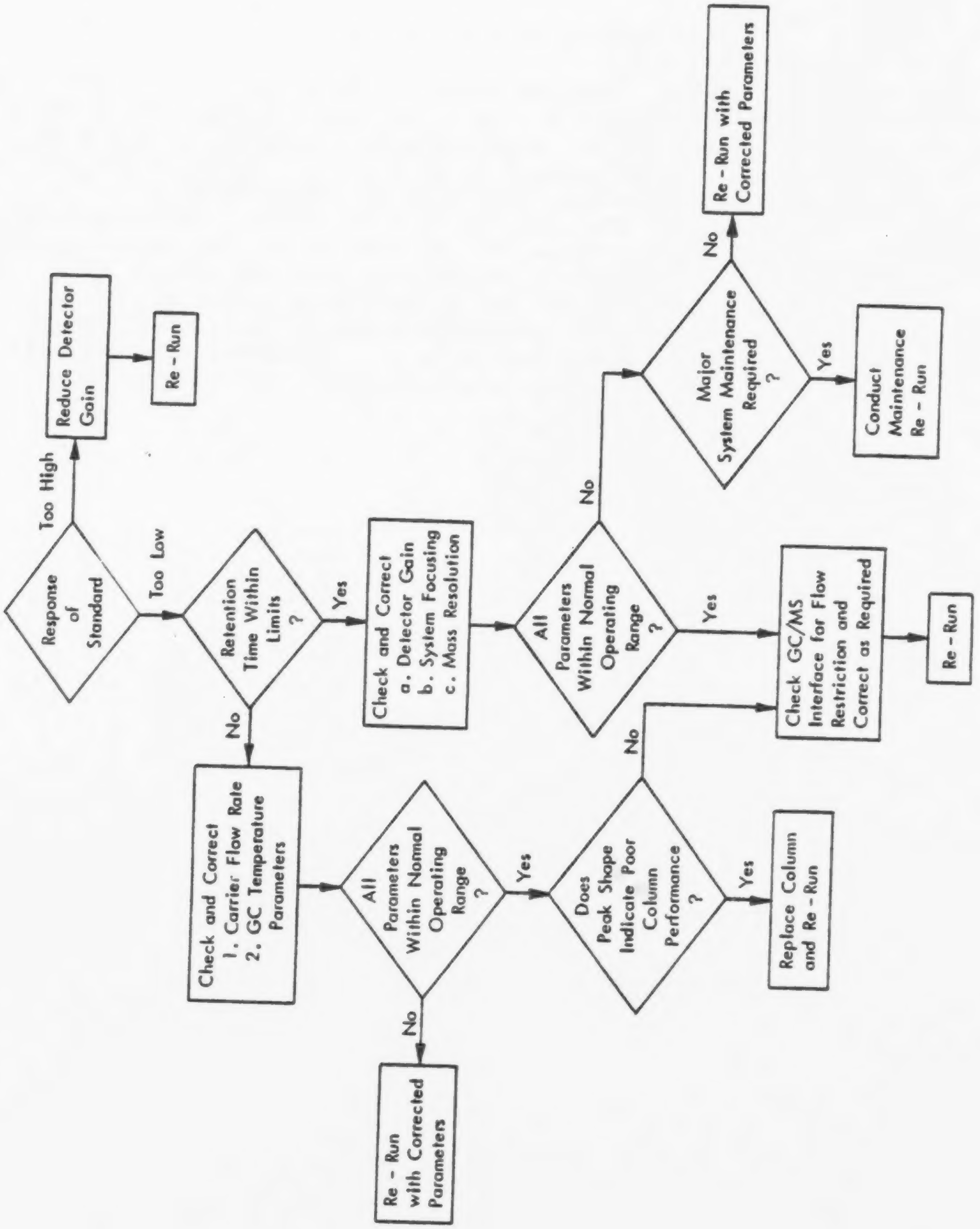

Response of Standard
Too High
Reduce Detector Gain
Re - Run
Too Low
Retention Time Within Limits ?
Yes
Check and Correct a. Detector Gain b. System Focusing c. Mass Resolution
All Parameters Within Normal Operating Range ?
No
Major System Maintenance Required ?
No
Re - Run with Corrected Parameters
Yes
Conduct Maintenance Re - Run
Yes
Check GC/MS Interface for Flow Restriction and Correct as Required
Re - Run
No
Check and Correct 1. Carrier Flow Rate 2. GC Temperature Parameters
All Parameters Within Normal Operating Range ?
No
Re - Run with Corrected Parameters
Yes
Does Peak Shape Indicate Poor Column Performance ?
No
Yes
Replace Column and Re - Run

Figure 4 - Flow Diagram for GC/MS Corrective Action

Appendix Q
Plan 4.11

Wright State University

QC Plan

QUALITY ASSURANCE PLAN FOR ANALYSIS OF SAMPLES FROM LOVE CANAL

1.0 Project Description and Organization.

Wright State University/Brehm Laboratory is functioning as a subcontractor to GCA/Technology Division which is working under prime Contract No. 68-02-3168 with the U.S. Environmental Protection Agency to assess the levels of selected environmental pollutants in the region of the Love Canal in Niagara Falls, New York. The function of Wright State/Brehm Laboratory is to accomplish analyses of tetrachlorodibenzo-p-dioxins (TCDDs) in soil, sediment, water, and air samples from the Love Canal area using analytical procedures developed by Wright State. These methods are capable of detecting parts-per-trillion levels of these compounds, and can also provide information on the isomeric composition of TCDDs present in the samples.

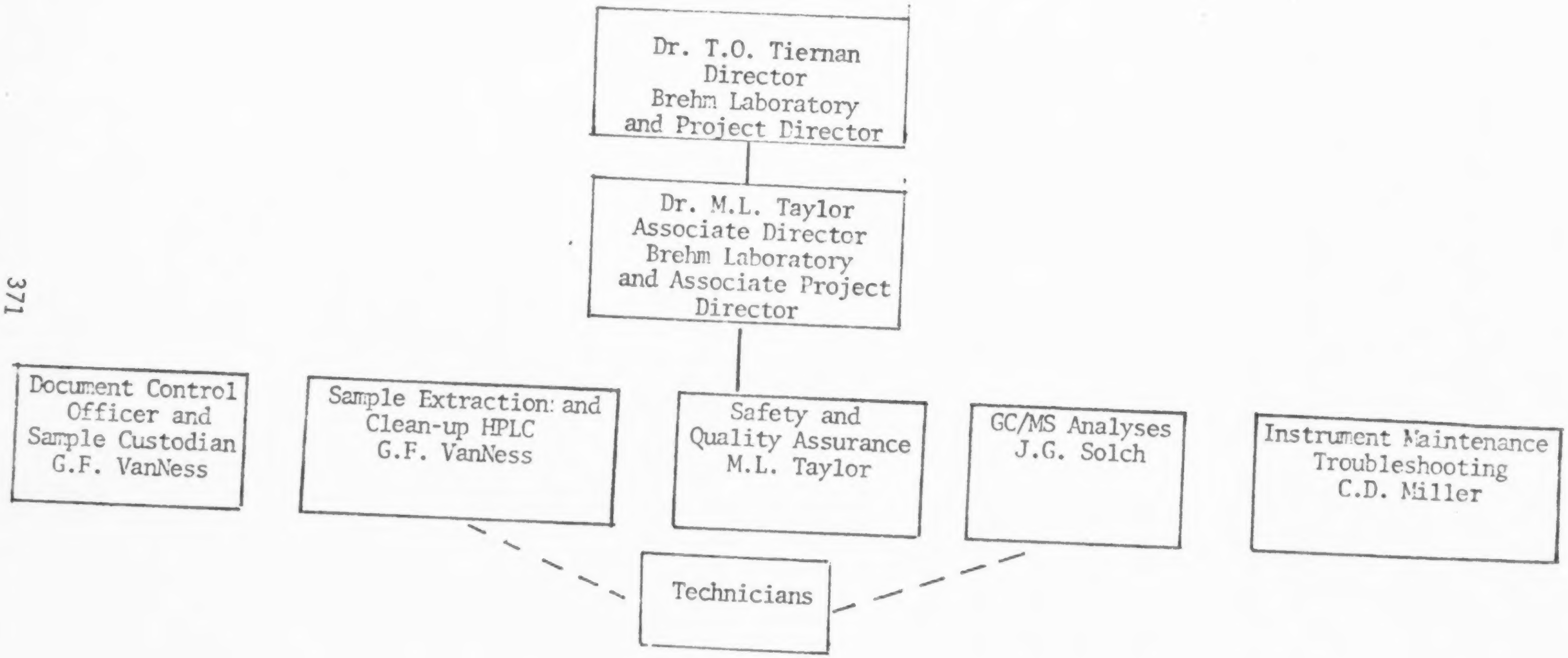
Wright State's work on this program is under overall technical and managerial direction of Dr. Thomas O. Tiernan, Director of the Brehm Laboratory. An organization chart showing the various tasks associated with this program, and identifying the responsible individuals for each phase of the program is attached.

2.0 Major Project Elements.

The major elements of Wright State's project conducted under this subcontract include:

WRIGHT STATE UNIVERSITY
LOVE CANAL STUDY - SUBCONTRACT NO. 1-619-026-222-007A
TO GCA CORPORATION - TECHNOLOGY DIVISION PRIME
CONTRACT NO. 68-02-3168 WITH
U.S. ENVIRONMENTAL PROTECTION AGENCY

WSU/BREHM LABORATORY - PROJECT ORGANIZATION


Dr. T.O. Tiernan
Director
Brehm Laboratory
and Project Director
Dr. M.L. Taylor
Associate Director
Brehm Laboratory
and Associate Project
Director
Document Control
Officer and
Sample Custodian
G.F. VanNess
Sample Extraction and
Clean-up HPLC
G.F. VanNess
Safety and
Quality Assurance
M.L. Taylor
GC/MS Analyses
J.G. Solch
Instrument Maintenance
Troubleshooting
C.D. Miller
Technicians

A) Establishment of internal document control and chain-of-custody procedures for samples.

B) Receipt of samples from the prime contractor (GCA) and entry into the system established in A).

C) Verification (by analyses of Wright State-spiked samples containing known quantities of TCDD) of the applicability of previously developed Wright State extraction and clean-up procedures for each category of sample to be analyzed; development and implementation of modifications to these procedures where necessary, and verification of new methods.

D) Analyses of actual samples using verified methodology.

E) Implementation of an internal Quality Assurance program to ensure that acceptable precision and accuracy are maintained throughout the analyses.

F) Performance evaluation by analysis of EPA-spiked samples received from the prime contractor.

G) Establishment and implementation of data reporting procedures.

3.0 QA Objectives

Analytical methods which are applicable for the determination of TCDDs in samples such as those to be collected in this study are in a continuing state of development. Wright State has previously developed and applied analytical procedures for quantitating TCDD at levels as low as parts-per-trillion in samples similar to those anticipated in the present project, but experience indicates that such methods must be validated for virtually every type of sample processed. This is accomplished, in each case, by spiking samples of a given type (sediment, water, etc.) with a known quantity of $^{37}Cl_4$-2,3,7,8-TCDD prior to processing and determining the recovery of this spike after applying the extraction and clean-up methods which are expected to be effective. Wright State's previous experience in analyzing many types and numbers of samples for TCDD has shown that recoveries of the $^{37}Cl_4$-2,3,7,8-TCDD in the range of 80-100% can usually be achieved by appropriate optimization of the extraction and clean-up procedures. In general, however, the methods are deemed to be acceptable if 50% or more of the added labelled TCDD spike is recovered. Since the quantitation of the native TCDD in the sample is always accomplished by reference to the $^{37}Cl_4$-2,3,7,8-TCDD internal standard which is added in known quantity (and any loss of $^{37}Cl_4$-2,3,7,8-TCDD in processing and analyses is assumed to reflect proportionately the loss of native TCDD present in the sample), the absolute recovery of the spike is not critical. In some cases samples which are very heavily contaminated with other chemical residues exhibit less than 50% of the added labelled TCDD spike. Again, this does not necessarily adversely affect the accuracy which can be achieved in quantitating native TCDD in the sample, but usually poorer recoveries result in higher minimum limits of detection for TCDD than are possible when better recoveries are achieved.

The precision and accuracy which can be expected for TCDD determinations in samples such as those encountered in this study using the methodology developed by Wright State must be ascertained on the basis of the results of analyses of samples spiked with known quantities of TCDD. In order to obtain an objective assessment from such analyses, the analysts should not know the quantities of the added spike until analysis is complete. To date, an insufficient number of native-TCDD spiked samples (at the ppt level) of the type encountered in the Love Canal study have been analyzed to permit a statiscally valid estimate of the precision and accuracy which can be achieved. For the spiked samples which have been analyzed, the precision is typically on the order of ±30%, and the accuracy is such that the TCDD levels determined agree with the levels added to the samples within ±30-50%. However, the variability which can be expected for analyses of samples such as the Love Canal samples is likely somewhat greater owing to the following factors:

A) Variability in Sample Composition. Even among samples of a given type, such as sediments, enormous variability of the samples, in terms of total chemical composition, is possible and even likely. This is particularly true of sites where many different types of chemical compounds have been deposited and subjected to natural mixing and dispersion forces over a period of years. Since the efficiency of the extraction and clean-up procedures for TCDD is markedly dependent upon the types and quantities of other chemical residues in the sample, this variability in sample composition can be expected to introduce substantial variability in the analytical data for TCDD.

B) Sample Inhomogeneities. Samples of the type encountered in this study are notoriously inhomogeneous and it is virtually impossible to select a truly representative aliquot of the samples for analysis. (It is also generally impractical to analyze the entire sample). Sediments for example, may contain a wide variety of types of soil, sand, gravel and other matrix materials, in addition to the chemical constituents. These are rarely uniformly distributed throughout the sample and true homogenization is impossible. Similarly, water samples frequently contain sediments which cannot be uniformly dispersed throughout the sample, even upon agitation. In both instances, compounds such as TCDD may be preferentially bound on certain sample components (for example, finely dispersed particulate in the water samples or on particular types of soil, etc.). All of these factors lead to substantial variabilities in analytical precision and accuracy.

C) Extraction Efficiencies. The true extraction efficiencies for removing TCDD from different types of sample matrices are difficult to establish, since there is evidence that compounds such as TCDD may bind very strongly (even irreversibly) to some types of particulates, soils, or sediment constituents. The extraction efficiency determined on the basis of a TCDD spiked sample does not necessarily correspond to that applicable for a sample in which TCDD is incorporated into the matrix, and is at best an approximation for many samples of the type analyzed here.

In view of the above uncertainties, the TCDD data obtained in this study may exhibit variability which is in the range of ±100-300%.

4.0 Document Control Procedures.

Samples received by the Brehm Lab in connection with this study are assigned Brehm Lab control numbers (beginning with GCA-1 and continuing serially) and these are entered into a Log Book maintained for this purpose, along with the corresponding GCA-assigned sample numbers. The Brehm Lab Document Control Officer, Mr. Garrett VanNess, makes all control number assignments and Log Book entries. He also prepares an internal memo form, containing information on the numbers, types, and control designations for each batch of samples, as they are received from GCA, and transmits this to the Project Director, Dr. Tiernan, who maintains this record in a central file for the project. Custody forms, received with each sample shipment from GCA, are also transmitted by the Document Control Officer to the Project Director and are also maintained in a secure central file. Sample tags attached to the sample vessels received from GCA are kept with the samples until final disposition. The control number assigned to each sample is used on all instrumental data records, lab book notations, and data sheets relevant to that sample. Data sheets summarizing the results of analyses for each batch of samples analyzed are initially prepared by the Document Control Officer, in conjunction with the GC-MS analyst, and are transmitted to the Project Director, who maintains these in a central file.

5.0 Chain of Custody.

The Brehm Lab sample custodian assigned for this project is Mr. Garrett VanNess. Mr. VanNess receives and retains in his possession at all times the key which opens the locked sample shipment containers which are received from GCA. These containers are delivered to Wright State by the air carrier, Federal Express, and arrive at Wright State's Shipping and Receiving Office. The containers, as received, are immediately transported by Wright State personnel to the Brehm Laboratory, where they are accepted by Mr. VanNess. Mr. VanNess unlocks the sample containers, checks any seals to ensure that they are intact, removes the samples and examines their condition. He then prepares an internal memo form which lists the samples received by number (both the Brehm Lab number which he assigns and the GCA-assigned number), the condition of the samples, the source of the shipment, the date received and any other pertinent information. Mr. VanNess signs the custody form which accompanies each sample shipment, acknowledging receipt by the Brehm Lab, and transmits the custody form and the internal information memo on the samples to the Project Director, Dr. Tiernan. The Project Director places these forms in a secure central file. Mr. VanNess then transfers the samples with the tags in place on each sample vessel, to a locked storage cabinet. Only Mr. VanNess and the Project Director possess keys which open this cabinet. All samples remain in lockage storage cabinets until they are analyzed. At the time of analysis, each sample is removed from storage, and transferred to the laboratory, where a portion of the sample is removed and analyzed. The sample documentation number is affixed to all sample vessels, lab notebook notations, data records, etc., so that the identity is clearly maintained. After removing an aliquot of a sample from the shipping vessel, the latter is again returned to a locked storage cabinet where it remains till final disposition, or until further analysis is required. Access to the samples is restricted to authorized personnel who accomplish the analyses. Only these personnel are permitted within the laboratories in which sample processing and analysis occur.

Portions of some of the sample extracts prepared by the Brehm Laboratory are shipped to Mr. Robert Harless, EPA/HERL, RTP, North Carolina. In order to maintain a custody record for these extracts, a Brehm Laboratory form is used which accompanies the extracts shipped. This form is signed by the Brehm Lab Sample Custodian, Mr. VanNess, and upon receipt at RTP, by Mr. Harless. Space is also provided on the form to indicate a list of the sample numbers (which are encoded by Brehm Lab), the shipper, the date of shipment, the date of arrival at RTP, and the condition of the samples. Upon receipt and acceptance of custody of the sample extracts, Mr. Harless signs the custody form and returns it by mail, to the Brehm Lab Project Director, who maintains this record in a central file with other records for the program.

6.0 Analytical Internal QC Checks.

The procedures applied internally by the Brehm Lab to effect quality control are essentially the same for all media analyzed in this program (soil, sediment, water, air filters). Prior to actual analysis of any group of samples of a particular type, the methodology which has been developed by Wright State is tested and optimized by analyzing a series of control samples, which are of essentially the same sample matrix type (soil, sediment, etc.) to which known quantities of TCDD have been added. These samples are spiked by personnel other than those involved in the actual analysis, and the analysts do not know the level of TCDD added to the sample until after the results of their analysis have been reported. These results are presented to the Project Director or Associate Project Director, who reviews the results with the analytical team and decides if the method is acceptable. Generally, for samples such as those analyzed in this project, the method is deemed acceptable if 70-100% of the added TCDD is recovered and detected in the analysis. If this is not the case, then modifications are made to this method to further optimize the performance, and additional spiked samples are analyzed using the revised procedures. This is repeated until a satisfactory level of performance is obtained for the spiked samples. Subsequently, this procedure is applied to analyze actual samples of the type for which methodology has been verified. This verification procedure is utilized for each type of sample to be analyzed in the project. Some additional TCDD-spiked quality control samples are also analyzed along with actual samples in the course of the project, in order to assure quality on a continuing basis. These data are reviewed regularly by the Project Director or Associate Project Director, and if at any time, the results for QC samples fall outside the range of acceptability (generally a recovery of 70-100% of the spike is desirable), then an entire new series of QC analyses is repeated, and the methodology is thoroughly checked to determine the source of error. Selected Love Canal samples analyzed during any period when unacceptable QC data are obtained are also reanalyzed to ensure that these were not also affected by any temporary aberrations in the in the methods or procedures.

An additional measure of quality assurance is provided in this program by the provision for a repeat analyses of Brehm Lab sample extracts by the EPA/HERL(RTP) laboratory. Extracts of selected samples in which TCDD is detected by the Brehm Lab are shipped to Mr. Robert Harless at this RTP laboratory for confirmatory analysis.

7.0 Data Reporting Management System.

TCDD analytical data obtained by the Brehm Laboratory in this project are reported in a format which is customarily used in other EPA-sponsored TCDD programs and which is unique to this determination. These forms are provided by the Brehm Laboratory. A sample form, showing tabular headings is attached.

The procedures applied within the Brehm Lab for data reporting are as follows. Upon completion of each set of analyses, the senior analysts (J.G. Solch and G.F. VanNess) tabulate the results and provide these data to the Project Director (Dr. Tiernan) and the Associate Project Director (Dr. Taylor). The latter review each batch of data as received, check it for consistency and accuracy, and then either the Director or Associate Director reports each set of data by telephone to the GCA Technical Monitor for the project, Dr. Robert Bradway. This is followed by preparation of an interim written report of the data (again by either the Director or Associate Director) and this report is forwarded to GCA (Dr. Bradway).

8.0 Corrective Action Procedures.

Corrective action procedures are implemented by the Senior analysts and other staff at the direction of the Project Director (Dr. Tiernan) and/or Associate Project Director (Dr. Taylor), who continuously monitor progress on the project and review the data as it is obtained. Biweekly meetings are held with the analytical staff to discuss any problems which have arisen, to review analytical results, and to clarify scheduling and reporting requirements.

TCDD DATA REPORTING FORM

WRIGHT STATE UNIVERSITY - BREHM LABORATORY RESULTS OF ANALYSES OF LOVE CANAL SAMPLES FOR TETRACHLORODIBENZO-p-DIOXIN USING GAS CHROMATOGRAPHY-HIGH RESOLUTION-MASS SPECTROMETRY

WSU Sample Number	GCA Sample Number	Native TCDDs Found (ng/g)			Percent Recovery of $^{37}Cl_4$-2,3,7,8-TCDD Internal Standard	Minimum Detectable Concentration (ng/g)
		m/z 320	m/z 322	Average		

Advanced Environmental Systems, Inc.
QA Plan

QA Plan for TOC, TOX, pH, and Conductance

1.0 Project Description and Organization

Water samples taken from Love Canal are analyzed for TOC, TOX, pH, and Conductance.

2.0 Major Project Elements

A. TOX - Determination of Total Organic Halide

B. TOC - Determination of Total Organic Carbon

C. pH - Determination of pH of sample

D. Conductance - Determination of specific conductance of sample

3.0 QA Objectives

4.0 Document Control Procedures

5.0 Chain of Custody - Same as 4.0

6.0 Analytical Internal QC Checks

7.0 Data Reporting Management System

8.0 Corrective Action Procedures

A. Determination of Total Organic Halide (TOX)

The method for the determination of total organic halide (TOX) in water utilizes the Dohrmann DX-20 TOX Analyzer system. This system is designed to concentrate organic matter in water onto granular activated carbon which is subsequently analyzed for TOX by microcoulometry.

The detection limit of this method under ideal conditions is 1 ug/l of organic halides. Organic halides are defined as all organic species containing chlorine, bromine, and iodine. Fluorine containing species are not determined by this method.

The procedure consists of the following steps:

1. Adsorption of Organics onto Granular Activated Carbon

   Approximately 40 mg of granular activated carbon (GAC) is placed in a clean, dry glass microcolumn. The column is connected to an adsorption apparatus. A water sample, which has been treated with sulfite to reduce residual chlorine to chloride and acidified to pH=2, is forced through the GAC column by pressurizing the sample reservoir. One hundred twenty-five ml of sample is usually required.

2. Nitrate Wash Step

   The GAC column is transferred to the wash channel of the adsorption apparatus and flushed with nitrate wash reagent to remove inorganic halides.

3. Combustion Step

   The GAC is removed from the glass column and placed in the boat inlet of the analyzer module and the TOX cycle initiated. The quartz boat containing the carbon moves into a high temperature furnace where organics are oxidized and halides are converted to the corresponding hydrogen halide (HX).

4. Coulometric Titration

   The halides then flow into a microcoulometric titration cell where the HX removes silver ions by precipitating AgX. The silver ions thus consumed are replaced electrochemically, and the generating current is integrated and displayed digitally as micrograms of chlorine.

B. Determination of Total Organic Carbon (TOC)

The method for the determination of total organic carbon (TOC) in water utilizes the Oceanography 600 TOC Analyzer. This system is designed to measure TOC through combustion of the sample. The sample is injected into the injection module which consists of an oxygen purification train, a combustion train, and an acidification train for inorganic carbon. Carbon is converted to carbon dioxide gas which is analyzed with an infrared detector.

The procedure consists of the following steps:

1. Oxygen Purification Step

   Oxygen carrier gas is passed through the sample to purge the sample of carbon dioxide.

2. Combustion Step

   The sample enters a combustion tube where it is volatilized in the purified oxygen stream to carbon dioxide. Carbon - containing compounds which are volatilized but not converted to CO_2 are swept into the high temperature combustion furnace by the oxygen carrier gas, and quantitative conversion to carbon dioxide occurs.

3. Acidification Step

   The acidification train consists of an acid chamber with an injection port and a drainable water trap which are separated by a glass frit. The oxygen stream and any water from the combustion train passes into the water trap and streams through the frit into the acid chamber. Inorganic carbon injected into the chamber is converted to carbon dioxide and purged from the acid by the oxygen.

C. pH Measurement

The Select Ion 5000 Ion Analyzer is set to measure pH and calibrated for two standards that emcompass the range of interest.

An ion sensitive electrode is placed in the sample and the digital pH readout is recorded.

D. Conductance Measurement

The Barnstead Conductance Meter is calibrated using a standard KCl solution. A 1 cm conductance cell is put into the sample and the specific conductance is recorded by adjusting the indicator needle to zero.

3.0 QA Objectives

A. Precision

A duplicate analysis is performed with every 10 analysis or with each run, whichever is less frequent.

The range of the duplicate analysis is calculated and compared to critical range tables developed for each parameter.

Should the range of the analysis fall outside the critical range value, the analysis is "out of control" and the run is repeated.

B. Accuracy

Spiked or known samples are run with every 10 analysis or with each run, whichever is less frequent.

The percent recoveries of the spiked analysis are calculated. If the recoveries are not within the control limits ($\overline{X} \pm 3SD$, where $\overline{X}$ = mean percent recovery and SD = standard deviation) established by the control chart, the analysis is "out of control" and repeated.

4.0 Document Control Procedures

Document Control Officer: Judy McDougall

Included are a sample tag, an explaination of the flow of samples and chain of custody including our system of logging samples, a sample history sheet, and an example page from our master log.

5.0 Chain of Custody

Same as 4.0.

Advanced Environmental Systems
(716) 731-3291

Job# ______ Date ________ Time _____

Sample # _____ Analysis _ _ _ _ _ _ _
SP OP MS GC AA CL SC

Parameters: ______________________________

Taken by ____________ Witness ____________

Contact Phase	Customer Contact- Notes to be kept by sales-person. Should an order result, these notes are transferred to the Contract Folder.
Order Phase	A formal proposal should go out on each order. Contract letters assigned. P.O/Proposal/ Customer contact notes go to Contract Folder.
Sample Receipt Phase	Site history sheets are matched with sample history sheets which are then matched with sample containers. (Note: Site history sheets are only parpared when the samples are collected by AES. If the customer does not fill out a sample history sheet, the sample coordinator will fill one out at time of receipt
Sample "Log in" Phase (A)	Log in sample in the "Master Log". Prepare a "sample flow folder". This folder will contain the sample history sheet and a "Protocol outline sheet". Note: if there are site history sheets they go to the Contract Folder. Sample is assigned AA & A# code.
Inventory Control Protbcol Outline Phase (A)	Samples are loged into inventory. Protocol is outlined using the proposal in the contract folder as a reference. Samples are placed in storage. Sample assigned XO code. Sample Flow Folder is returned to sample coordinator.*
Sample "Log In" - Phase (B)	Sample Master Log # recorded in full. Folder transferred to Operations Manager for assignment of priority.
Priority Phase	Priority is assigned by Operations Manager. Folder forwarded to appropriate analyst. Note: Chain of custody for folders is kept by the analyst in his notebook. Sample coordinator uses notebook pages copied to track samples in master log*.
Analytical Phase	Sample removal from storage. Analytical routine begins. Folder and sample become the responsibility of analyst. Note: Operations Mgr re-prioritizes on daily basis. Analytical data is recorded on sample history as well as in lab notebook. When analytical scheme is complete sample flow folder is returned to sample coord.
Report Preparation Phase	Sample flow folder is reviewed. Notebook pages are colated with Sample History Sheet and Protocol Outline Sheet. All numbers are compared. Tables of data are drafted by typist. Drafted tables plus comple Sample Flow Folders are presented to Operations Mgr for preparation of report.
Report Phase	Once the report is prepared and forwarded, the report goes to the contract file along with all information contained in the Sample Flow Folders.
Inventory Control Phase (B)	The status of the remaining sample (if any) is reviewed.

* If sample requires split sample, history is prepared for each split Note that on the sample history associated with the original sample; under the comments section, the number of splits should be outlined. Sample history sheets associated with split samples are returned to sample coordinator for assignment of AES number.

CUSTOMER SAMPLE #	ALPHA/ ALPHA	ALPHA NUMBER	LAB CODE						ORG PREP		GC/MS		GC		AA		CLASSICAL		SUB-CONT		SAMPLE REC'D	DATE DUE	DATE COMPLETE	REPORT DATE
			OP	MS	GC	AA	CL	SC	IN	OUT	IN	OUT	IN	OUT	IN	OUT	IN	OUT	IN	OUT				

6.0 Table of QC Check for Operations

Analytical Method	Operational Checks (water)
TOX	Electrolyte titration solution checked Sample boat is changed once per day Gas pressure is monitored at the proper level GAC columns packed properly
TOC	Standard Curve constructed daily. Gas flow checked and all tubing connections checked for leaks EPA Test sample analyzed with each run. Sample must fall within 95% confidence limits.
pH	Calibration with each run using pH 7 and pH 10 buffer Electrodes washed with distilled deionized water after every sample. Calibration is rechecked with both buffers after every 10 samples.
Conductance	Calibration with Standard KCl solution. Wash electrodes after each sample. Check calibration after every 10 samples are run.

7.0 Data Reporting Management System

Does not apply.

8.0 Corrective Action Procedures

It is the responsibility of the individual analyst to routinely monitor the precision and accuracy of each analytical procedure.

Should the precision or accuracy checks as described in Section 6, demonstrate that the analysis is out of control, it is the analysts responsibility to repeat the analysis after closely scrutinizing possible sources of error.

After a discussion with the lab manager regarding possible sources of error, the analysis is repeated.

Instructions: Separate Sheet for Every Sample

II. Sample History

Order Code ______________________

AES Sample # ______________________

Customer Sample # ________________ Date Collected ________________________

Analyses ____

Container ____

Vol. submitted ____

Sample Number	Relinquished By:	Received By:	Time	Date	Reason for change of Custody

Comments/Footnotes: ____

Analytical Results: ____

Appendix Q
Plan 4.13

Wadley Institutes of Molecular Medicine

QA Plan

1.0 PROJECT DESCRIPTION AND ORGANIZATION

Wadley Institutes of Molecular Medicine is under contract to GCA/Technology Division to test approximately 100 live mice to determine the ability of their cells to produce Type 1 Interferon. The mice will be collected in the Love Canal area of Niagara Falls, N.Y., and shipped live to Wadley.

Dr. Amanullah Khan will direct the project work with John Duvall serving as Project QA Coordinator, Sample Custodian and Document Control Officer.

2.0 BIOASSAY PROCEDURES

Mouse cells are induced to produce interferon by the induction procedure given below and then the interferon produced is assayed by a modification of the technique described in Reference 1.

The mice are shipped live with food in the cage.

A. Induction Procedure - Type 1 Interferon

1. Mouse spleens removed by sterile techniques.
2. Spleen teased apart and cells washed.
3. Erythrocytes lysed by NH_4Cl.
4. Leukocytes washed in PBS-EDTA.
5. Cells counted and resuspended to 10^6 cells in MEM with 5 percent FBS.
6. 0.2 ml of cell suspension added per well of a 96 well sterile microtiter plate. One row of 12 wells inoculated/mouse.
7. Cells primed with marginal and optimal amounts of sendai virus and incubated 2 hours at 37°C.
8. Cells then induced with marginal and optimal doses of sendai.
9. Four wells of each row of cells receive media only to check for spontaneous production.
10. Plates incubated 20 hours overnight.
11. Cells supernatants harvested and assayed for amount of interferon produced.

B. Interferon Assay Procedure

1. Murine & L929 fibroblasts are grown to confluency in 96 well microtiter plates and fed the day before and the day of use with 150 µl of MEM 5 percent FBS. Before use cells are inspected for contamination and confluent growth microscopically.

2. Samples and standards (WHO International Reference Prep. mouse interferon 12,000 IU/ml prepared by National Institute of Allergy and Infectious Diseases, NIH, Bethesda, Maryland, USA, catalog (#G-002-94-511) are stored at -70°C until used.

3. Samples and standards are added to duplicate wells of plates, 1 standard and 4 unknown samples/plate. These are diluted 2 fold vertically down the plate.

4. Cell (+) and viral (-) controls are included on each plate--one row of 8 cells/plate for each control.

5. Cells with sample dilutions are incubated 20-24 hours at 37°C, 5 percent CO_2.

6. All media, sample, and standard dilutions are removed and VSV added to each well except the cell control which receives fresh media. Plates are incubated approximately 24 hours until 90 percent CPE of the viral control is produced in the viral control wells as determined by microscopic examination.

7. 150 µl of neutral red dye in media 5.5×10^{-5} gm/ml is added to all wells and the plates are incubated 1.5-2 hours.

8. Plates are then washed twice with saline and dye extracted from viable cells by the addition of 150 µl of acid ethanol to each well.

9. The developed plate is read spectrophotometrically on a Titertek Multiskan plate reader and readings fed into the computer for data processing. The titers of each sample are reported on a terminal and recorded, expressed in reference interferon units/ml.

C. MODIFICATIONS TO BIOASSAY

The following modifications to Reference 1 procedures are incorporated in the Section B procedures above.

1. Cell Line: a-L929 clone of mouse cell line ATCC CCl_1 NCTC clone 929.

2. Plate set-up: Samples were assayed in duplicate at 8 different 2 fold dilutions. NIH reference mouse interferon was included in duplicate on each plate of assays. This titer was used to compute the reference correction value.

3. Reading developed plates: Plates were read directly with a Titertek, Multiskan plate reader directly interfaced to the computer. All computations are done by the computer and data stored there.

Reference

1. DUVALL, KAHN, HILL, GROUND, MUNTZ, LANIUS, "Computer-Assisted Microdye Uptake Human Interferon Assay" in "Interferon Properties and Clinical Uses", KAHN, A., HILL, N.D., and DORN, G.L., editors. LELAND FIKES Foundation Press, 1980.

3.0 INTERNAL QC PROCEDURE

The quality control procedures used in this bioassay are briefly described below. All samples are analyzed both induced and non-induced and standard tissue QC techniques are followed for this in vitro test system. Table 1 summarizes the frequency, control limits and corrective action appropriate to these techniques.

A. Negative Controls (viral controls)

A negative control (virus challenged) of 8 wells is run for each group of the standard interferon preparations and 4 unknown samples. The values for these data are incorporated as the background of the assay and used in all calculations of titers. This represents 100 percent CPE.

B. Positive Controls (cell control)

Positive controls (cell control) of 8 wells are run for each group as above. This data represents the 0 percent CPE and is used in the calculation of all sample titers for that plate.

C. Reference Standards

WHO International Reference Preparation of Mouse Interferon, 12,000 IU/ml prepared by National Institute of Allergy and Infectious Diseases, NIH (catalog #G-002-904-511) is run in duplicate at 8 dilutions. It is assayed with each group of 4 unknown sample titers obtained from the reference standard and used to compute correction factor for the unknown samples to yield reference units.

D. Replicate Assays

All standards and unknown samples are run in duplicate and raw data is averaged to calculate titers.

4.0 CORRECTIVE ACTION PROCEDURE

The project QA Coordinator, John Duvall, is responsible for the implementation of corrective action whenever necessary. Table 1 gives control limits for operation checks and the planned corrective action to be taken if these control limits are exceeded. The procedure for documenting corrective action is that all assay data are stored in the computer banks for reference, including re-assayed samples.

TABLE 1. INTERNAL QC REQUIREMENTS - TYPE 1 INTERFERON ASSAY

Operation check	Frequency	Control limits	Corrective action planned
Negative Controls	Each group of 4 samples	85-95% CPE	Rerun standard plus samples
Positive Controls	Each group of 4 samples	Not applicable	Not applicable
Standards	Each group of 4 samples	Standard assays within half to twice its known value	Rerun standard plus samples
Replicate Assays	Every sample	Twice the variance of well to well replicates	Edit dilution from calculations Reassay sample

5.0 GEOTECHNICAL STUDY

The subcontractors involved in the geotechnical study are listed below. The suggested QA Plan outline provided to these subcontractors is presented on the following page.

Subcontractor	Project Activity
JRB Associates, Inc.	Supervisory geologist-interpretation of existing geotechnical information, coordination of drilling program, geologic logging, collection of groundwater-related samples
Empire Soils Investigation, Inc.	Well drilling
Technos, Inc.	Geophysical surveys
GeoTrans, Inc.	Geophysical modeling

Subcontractor's
QA Project Plans
for
Geotechnical Services at Love Canal

Suggested Outline

1.0 Project Description and Organization - include organization chart naming key personnel.

2.0 Major Project Elements.

3.0 QA objectives - Identify for the appropriate project elements.

4.0 Document Control procedures - Overall framework, numbering system, etc. has been provided by GCA and described in Section 4.0 of their QA Plan. State your internal operation of the system, identifying your Document Control Officer.

5.0 Chain of Custody Procedures - Overall framework, sample tags, custody forms, etc. are provided by GCA. State your internal operation of the system, identifying the person responsible for custody procedures.

6.0 Project - Specific Internal QC Procedures - State the means you will use to monitor quality including operational checks on instruments when appropriate. Control limits for each operational check should be provided with the corrective action to be taken if control limits are exceeded.

7.0 Data Reporting and Review - Internal procedures to be used should be described.

8.0 Corrective Action Procedures - These corrective actions should have been identified in Section 6.0. State how they will be implemented in your project activity - identify responsible personnel - and how they will be documented.

JRB Associates, Inc.
QA Plan

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1.0 PROJECT DESCRIPTION AND ORGANIZATION

1.1 BACKGROUND

GCA/Technology Division is currently under contract to the Environmental Protection Agency to evaluate pollutant levels present in the Love Canal Area of Niagara Falls, New York. The program involves a multifaceted sampling and analytical effort designed to detect and quantify a variety of trace metals, volatile organics, pesticides, and other compounds in soil, sediment, air, biota, surface water and groundwater samples. Pollutant flow in groundwater will be modelled using geophysical test data as well as hydrological test data from monitoring wells installed during this program.

The geotechnical investigations will be performed in two phases: Initial Test Phase and Monitoring Phase. The Initial Test Phase will begin in August. The Monitoring Phase will begin in September and run through October.

The Initial Test Phase will be conducted to describe the subsurface geology, occurrence of groundwater and location of contaminant plumes in the area. These activities will be conducted both in areas which are suspected of being contaminatd and those believed to be free of contamination. At least one test well shall be used as a check of drilling and sampling techniques. The results of these tests will be used to establish background or control conditions and to determine the locations of subsequent test sites.

Appropriate geophysical methods including but not limited to ground penetrating radar, seismic surveys, electromagnetic surveys, magnetometer surveys, and simple resistivity will be used to accomplish the geotechnical requirements of the Love Canal Monitoring Project. Two wells will be drilled (estimated depths 150 feet) through the Lockport Dolomite in an area believed to be free of contamination. NX core samples of the bedrock, in conjunction with resistivity well logs and hydrologic tests, will be used to establish stratigraphy and the occurrence of groundwater aquifers in the bedrock. Ten (10) pairs of test wells will be drilled in the initial test phase: one each pair to the top of the underlying glacial till (estimated depth 25 feet) and the other into the upper part of the underlying dolomite formation (estimated depth 60 feet).

The Monitoring Phase will include further geophysical surveys, groundwater modelling and the installation of one hundred ten (110) additional pairs of monitoring wells. Hydrologic testing of the wells and chemical analysis of groundwater samples will be performed. Data from the Monitoring Phase will accurately define the movement of groundwater in each aquifer as well as the location and characteristics of any plumes of contamination.

Drilling, geophysical surveys, and groundwater modelling will be accomplished under separate procurements.

1.2 JRB's SCOPE OF WORK

Activity I. Supervisory Geologist Services

The Supervisory Geologist Subcontractor shall be responsible for:

- Gathering and interpretation of existing geotechnical information
- Coordination of the drilling program with other program efforts
- Documentation of drill site selection
- Collection of a water sample from each of the wells
- Supervision of drilling, geologic logging, completion, and testing of two hundred forty two (242) wells, (see Table 1-1)
- Providing geophysical logs for two (2) deep wells (150 feet each).

Concurrent with other phases of this work, the Supervisory Geologist Subcontractor, with the assistance of the Prime Contractor, shall collect all existing information pertinent to the goals of the project from the New York State Departments of Health and Environmental Conservation and the U.S. EPA.

JRB's organization to provide the above geotechnical services to GCA is shown in Figure 1-1.

This information will be interpreted in such a way as to provide detailed guidance for the conduct of both the initial testing and monitoring phases of the project. Such interpretations will be concerned with, among other things: the number of aquifers encountered and the nature of their separation or interconnections; the proper location of test sites; and the manner of well completion which is most appropriate for existing conditions. The data interpretations of the Supervisory Geologist Subcontractor shall be discussed with the Prime Contractor prior to their use in directing or altering the geotechnical program.

The Supervisory Geologist Subcontractor shall coordinate the drilling program with other program efforts including geophysical testing and groundwater modelling. In conjunction with the Prime Contractor, the Supervisory Geologist Subcontractor shall use geophysical survey results, chemical analysis data, modelling results, modelling requirements, and other information to specify well placement.

The Supervisory Geologist Subcontractor shall document the basis for the selection of each drill site and report this information to the Prime Contractor on a weekly basis. Separate site-specific reports describing sources of information, data to be obtained, and the results of drilling activities shall be prepared for each drillsite.

The project will have very stringent requirements related to the drilling and construction of wells, coring procedures, and sampling of wells to avoid any possibility of introducing contaminants into any zone from external

TABLE 1. SCHEDULE OF WELL INSTALLATION

Time Period	Number of Wells	Estimated Depth (ft)	Comments
PHASE I			
August 30	2	150	Drilled into Lockport Dolomite; extract NX core samples taken into 10-foot sections
August 30	10	25	Drilled to top of till
August 30	10	60	Drilled into top of dolomite aquifer
PHASE II			
September - Ocotber 15	110	25	Drilled into the top of the till to establish monitor wells
September - October 15	110	60	Drilled into Lockport Dolomite

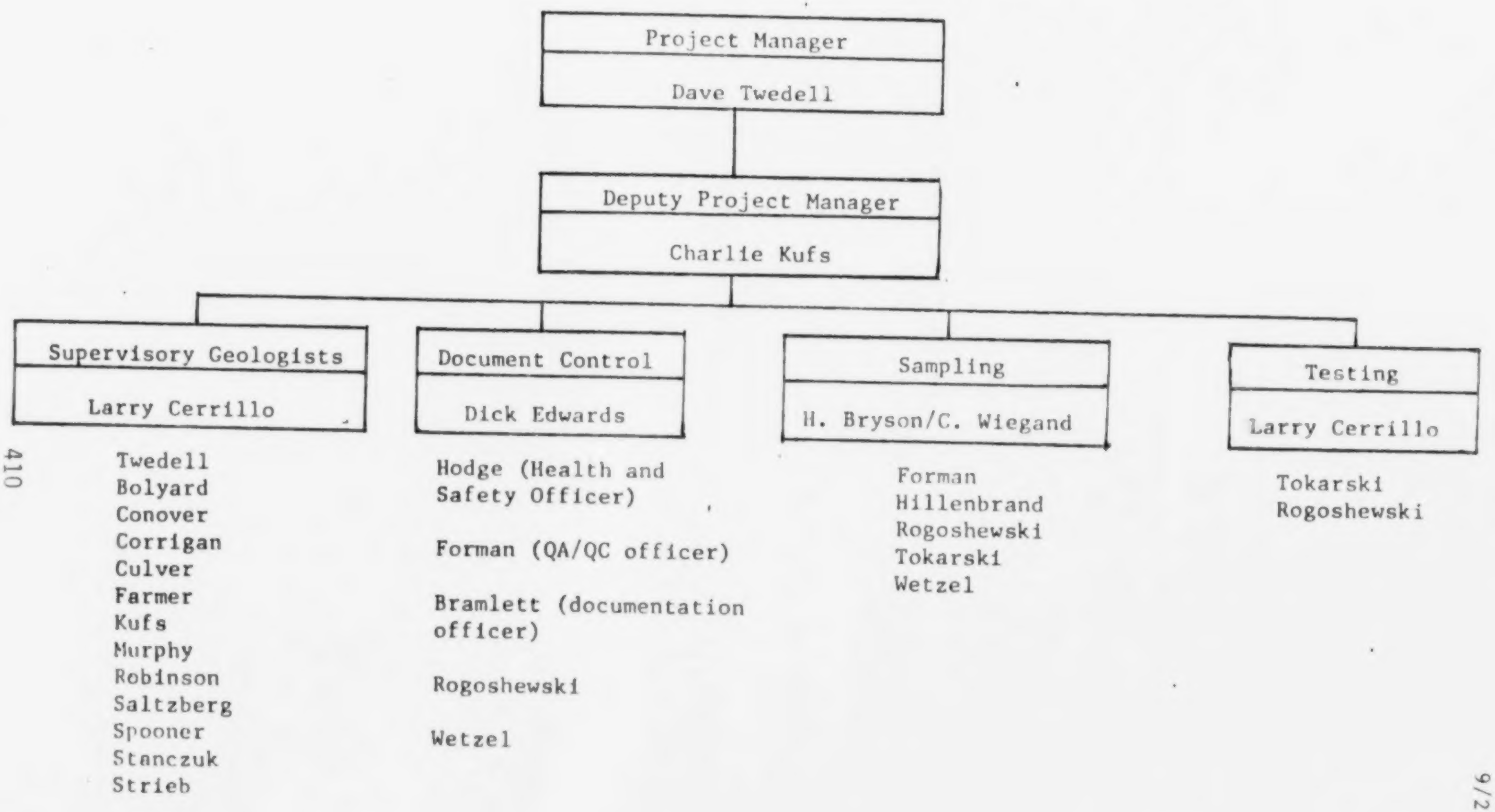
Project Manager
Dave Twedell
Deputy Project Manager
Charlie Kufs
Supervisory Geologists
Larry Cerrillo
Twedell
Bolyard
Conover
Corrigan
Culver
Farmer
Kufs
Murphy
Robinson
Saltzberg
Spooner
Stanczuk
Strieb
Document Control
Dick Edwards
Hodge (Health and Safety Officer)
Forman (QA/QC officer)
Bramlett (documentation officer)
Rogoshewski
Wetzel
Sampling
H. Bryson/C. Wiegand
Forman
Hillenbrand
Rogoshewski
Tokarski
Wetzel
Testing
Larry Cerrillo
Tokarski
Rogoshewski

Figure 1-1 JRB Organization for Geotechnical Investigations at Love Canal

sources. The Supervisory Geologist Subcontractor should review carefully the Statement of Work contained in the RFP for the drilling subcontractor (GCA 1-619-026-222-003A).

The Supervisory Geologist Subcontractor shall oversee the entire drilling program and be responsible for the geologic logging of each well. This includes providing a geologist at each drill site to continuously monitor drilling activity and geologically log all sediment and rock samples using acceptable descriptive terminology and classification systems (e.g., Unified Soil Classification System).

The Supervisory Geologist Subcontractor shall be responsible for performing all hydrological testing to establish the groundwater flow direction, gradients, aquifer permeability, and transmissivity. The methods employed to collect such data shall include but not be limited to water table measurements, pumping or slug tests.

The Supervisory Geologist Subcontractor shall collect all groundwater samples, sediment samples, and rock cores in appropriate containers and deliver them to the Prime Contractor. Proper methods for sampling as directed by the Prime Contractor shall be followed. A screening for TOC and TOX will be performed at the site by the Prime Contractor on an appropriate number of sediment and water samples.

The chain of custody as required by the U.S. EPA for all samples shall be maintained and be the responsibility of the Supervisory Geologist Subcontractor.

All quality assurance/quality control procedures as specified by the Prime Contractor shall be adhered to by the Supervisory Geologist Subcontractor. The project will have very stringent requirements related to the drilling and construction to wells, coring procedures, and sampling of wells to avoid any possibility of introducing contaminants into any zone from external sources.

2.0 PROTOCOL OBJECTIVES

The objective of this protocol document is to ensure the timely, safe, and technically valid completion of the required services. More specifically, this document is designed to provide the following:

- Written record of standard procedures
- Operating checklist for:
  - Technical
  - QA/QC
  - Safety and health matters
- A review document for independent critique and analysis
- Vehicle for promulgation of modifications and updates
- Ensure document control procedures are followed
- Ensure chain of custody procedures are followed.

3.0 MONITOR WELL DRILLING PROCEDURES

Introduction

The following procedures are designed to be used in the drilling of monitor wells in contaminated areas. The major objectives of the drilling program are to:

- Complete monitor wells so that samples can be taken
- Ensure that water producing zones and aquifers are sealed off from one another, thus preventing any cross-contamination, and
- Obtain accurate geologic and hydrologic information regarding the characteristics of these various water producing zones.

In addition, these procedures will be used for conducting formation testing to determine the flow and storage capabilities of the water bearing zone.

A. PRE-DRILLING SITE PREPARATION

Prior to the drilling of any groundwater monitor wells (GMW), a complete inventory of the site must be performed. This inventory includes:

- Obtaining owners written permission to drill
- Locating and verifying the existence of any underground utilities in the area
- Drawing a detailed sketchmap of the site, locating the hole for the drilling crew.

3.1 PERSONNEL

The requirements for personnel are as follows:

- 2 Field survey crewmembers
- 1 Drilling supervisor
- X representatives from each utility that may be affected (e.g., water, gas, electric, telephone, TV).

3.2 EQUIPMENT

Equipment requirements are as follows:

- 1 300-foot measuring tape
- 1 site sketch form
- 1 compass
- 1 clipboard and pen or pencil and waterproof marker
- 1 wooden stake with flagging
- 1 work authorization form
- 1 pipe locator.

3.3 PROCEDURE

1. Obtain a signed permission slip from the owner of the site prior to entering any property. NOTE: Even if prior entry has been granted, notify the owner of your activity each time you enter the site.

2. Have all representatives of the utilities affected mark, at the site, the location of pipelines that are on the property, and obtain a written report from them.

3. Stake out a potential drilling site with your wooden stake and flagging, making sure it will not interrupt any underground utilities.

4. Using the pipe locator, determine if any pipes are located beneath the potential drilling site. If any pipes are suspected, repeat steps 1.3.3 and 1.3.4 until a suitable site is found.

   STOP - If no suitable site can be located, report to the drilling supervisor for judgment.

5. Measure and record on the Sketch Map (see Figure 3-1) any permanent landmarks with a measuring tape. This step will ensure that the stake location will be recorded if the stake is lost. Mark site number on the stake with the marker.

6. Fill out the appropriate portion of the Work Authorization form (see Figure 3-2) and proceed to the next site.

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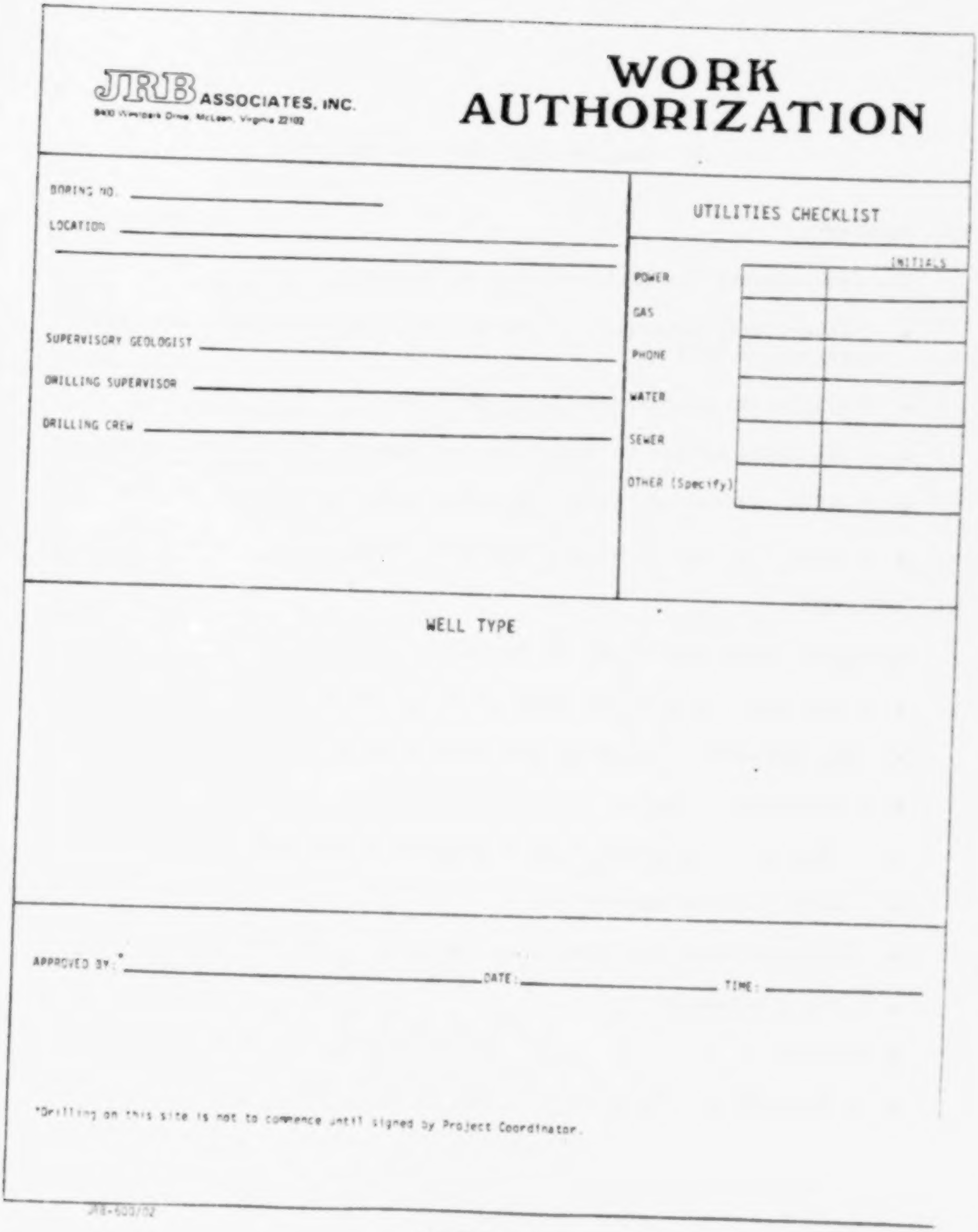
CHECK LIST:
BUILDINGS____
UTILITIES____
OTHER WELLS____
STREAMS____
ROADS____
SURFACE WATER____
ROCK OUTCROPS____
OTHER____

WELL NUMBER______ DATE______ SCALE________ MEASURED WITH____ NAME____________

SITE SKETCH

JRB-600/03-a

FIGURE 3-1.



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WORK AUTHORIZATION

BORING NO. ____________________

LOCATION ____________________

SUPERVISORY GEOLOGIST ____________________

DRILLING SUPERVISOR ____________________

DRILLING CREW ____________________

UTILITIES CHECKLIST

	INITIALS	
POWER		
GAS		
PHONE		
WATER		
SEWER		
OTHER (Specify)		

WELL TYPE

APPROVED BY:* ____________________ DATE: ______________ TIME: ______________

*Drilling on this site is not to commence until signed by Project Coordinator.

JRB-600/02

FIGURE 3-2.

B. SHALLOW WELL DRILLING PROCEDURE

3.4 PERSONNEL

The requirements for personnel are as follows:

- 1 supervisory geologist to oversee drilling operations and log progress of well
- 1 driller to conduct drilling operation
- 1 driller's helper to assist in the conduct of drilling operations
- 1 QC/QA Officer to conduct periodic inspection
- 1 safety officer to conduct periodic inspections.

3.5 EQUIPMENT

Equipment requirements are as follows:

- 1 drilling rig with all associated equipment
- required safety equipment (see Safety Manual)
- 1 conductivity meter
- 1 fiberglass measuring tape - weighted at the end
- 1 water soluble marking pen
- clipboard, well log form, pens, pencils
- 1 field notebook
- 1 watch
- 1 thermometer (C-scale).

3.6 PROCEDURES

1. Set up to drill over the wooden stake placed on the site by the survey crew.

   STOP - No drilling is to commence until a Work Authorization form is issued, and all personnel are wearing their safety equipment required in the Health and Safety Plan.

2. Begin to auger with a 6-inch diameter auger bit into the overburden.

3. As the drilling progresses, take samples of the overburden material each time it appears that a change has taken place in the physical composition of the drilled material. Record all descriptions of the overburden material onto the well log form (see Figure 3-3). NOTE: Be extra sensitive to any changes in the moisture content of the material as it is brought up by the auger bit. All cuttings and disposable safety equipment are to be disposed of in 55 gallon drums with lid until the area is declared clean.

4. When a suspected water bearing zone is encountered, stop drilling and measure for any water that may be in the hole. If water is not measured, continue drilling until either water or the glacial till is encountered.

5. If water is measured in the hole, continue drilling 2 additional feet.

6. Pull back the auger flight 1 foot, and install clay pellets.

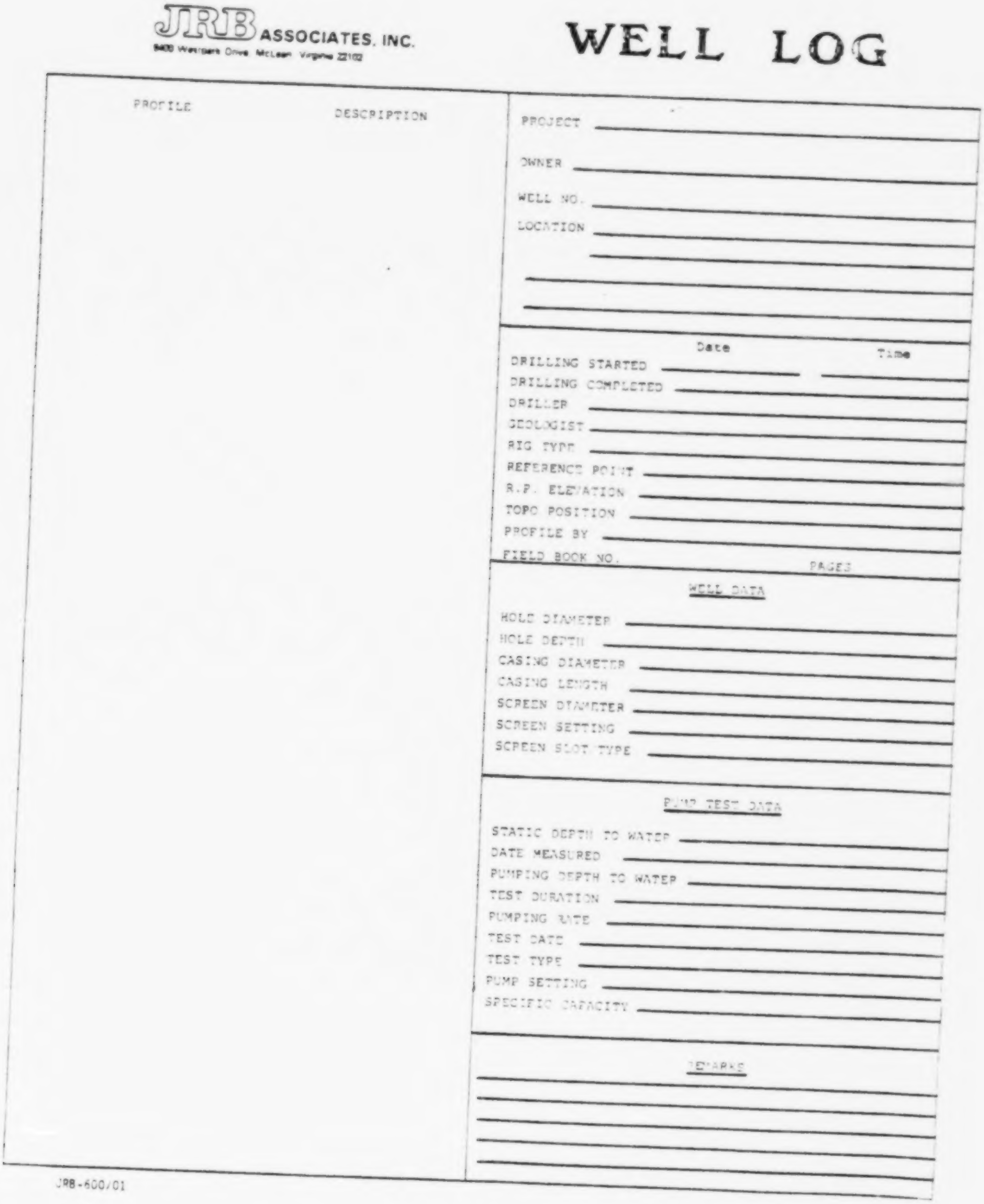
7. Withdraw auger flights from the entire hole.

8. Drive a 6-inch casing the total depth of the hole.

9. Install a 2-inch pipe into the hole and finish the well using the configuration shown in Figure 3-4.

10. Insure site is clear of all equipment and drums and that concrete culverts are installed properly before leaving site.

11. Place lock onto well cap and proceed to decontamination, following the procedure in Section 5 of this document.



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WELL LOG

PROFILE

DESCRIPTION

PROJECT ____
OWNER ____
WELL NO. ____
LOCATION ____

Date

Time

DRILLING STARTED ____ ____
DRILLING COMPLETED ____
DRILLER ____
GEOLOGIST ____
RIG TYPE ____
REFERENCE POINT ____
R.P. ELEVATION ____
TOPO POSITION ____
PROFILE BY ____
FIELD BOOK NO. PAGES

WELL DATA

HOLE DIAMETER ____
HOLE DEPTH ____
CASING DIAMETER ____
CASING LENGTH ____
SCREEN DIAMETER ____
SCREEN SETTING ____
SCREEN SLOT TYPE ____

PUMP TEST DATA

STATIC DEPTH TO WATER ____
DATE MEASURED ____
PUMPING DEPTH TO WATER ____
TEST DURATION ____
PUMPING RATE ____
TEST DATE ____
TEST TYPE ____
PUMP SETTING ____
SPECIFIC CAPACITY ____

REMARKS

JRB-600/01

FIGURE 3-3.

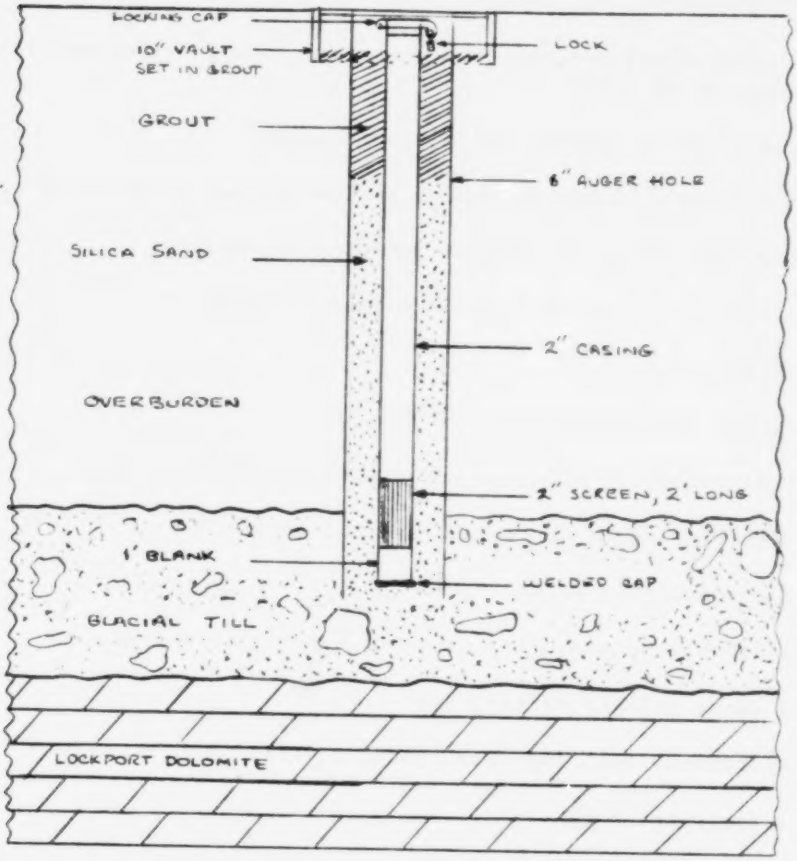
LOCKING CAP
10" VAULT
SET IN GROUT
LOCK
GROUT
6" AUGER HOLE
SILICA SAND
2" CASING
OVERBURDEN
2" SCREEN, 2' LONG
1' BLANK
WELDED CAP
GLACIAL TILL
LOCKPORT DOLOMITE

FIGURE 3-4.

TYPICAL WELL OVERBURDEN

C. SHALLOW BEDROCK WELL DRILLING PROCEDURE

3.7 PERSONNEL

The requirements for personnel are as follows:

- 1 supervisory geologist to oversee drilling operations and log progress of well
- 1 driller to conduct drilling operation
- 1 driller's helper to assist in the conduct of drilling operations
- 1 QC/QA Officer to conduct periodic inspection
- 1 safety officer to conduct periodic inspections.

3.8 EQUIPMENT

Equipment requirements are as follows:

- 1 drilling rig with all associated equipment
- required safety equipment (see Safety Manual)
- 1 conductivity meter
- 1 fiberglass measuring tape - weighted at the end
- 1 water soluble marking pen
- clipboard, well log form, pens, pencils
- 1 field notebook
- 1 watch
- 1 thermometer (C-scale).

3.9 PROCEDURE

1. Set up to drill over the wooden stake placed on the site by the survey crew.

   STOP - No drilling is to commence until a Work Authorization form is issued, and all personnel are wearing their safety equipment required in the Health and Safety Plan.

2. Begin to auger with a 6-inch diameter auger bit into the overburden.

3. As the drilling progresses, take samples of the overburden material each time it appears a change has taken place in the physical composition of the drilled material. Record all descriptions of the overburden material onto the well log form (see Figure 3-3). NOTE: Be extra sensitive to any changes in the moisture content of the material as it is brought up by the auger bit. All cuttings and disposable safety equipment are to be disposed of in 55 gallon drums with lids until the area is declared clean.

4. To verify the contact with bedrock, remove the center of the auger, replace with a split spoon and proceed to hit the rock until refusal is certain.

5. Once hard rock is verified, pull auger back 1 foot and install clay pellets.

6. Remove all remaining auger flights and drive a 6-inch drain casing firmly into bedrock.

7. Using a 5-1/2 inch tri-cone bit, wash the 6-inch casing clean of all material collected during the setting process. NOTE: All wash material being removed from the hole must be captured in a trough and pumped into a tanker truck for safe disposal.

8. Conduct gamma-ray log of the hole. NOTE: The gamma-ray log can be used to determine where the screened section of the shallow hole can be placed.

9. Based upon the instructions of the work authorization form, drill or core 5 feet into the bedrock. Careful measurement of drilling water used should be made, to enable an accurate determination of water bearing zones. In addition, conductivity and temperature readings should be taken at each 1-foot interval.

10. If water is encountered after drilling 5 feet, complete the well as shown in Figure 3-5.

11. If no water is encountered after drilling 5 feet, drill an additional 5 feet.

12. If water is encountered after drilling 10 feet, complete the well as shown in Figure 3-5.

13. If no water is encountered after drilling 10 feet, grout the 10-foot hole in the dolomite, pull the casing to within 2 feet below the upper water bearing zone or most likely water bearing zone, and complete the well as shown in Figure 3-4.

14. Once the well is installed, take final static water level prior to leaving the site.

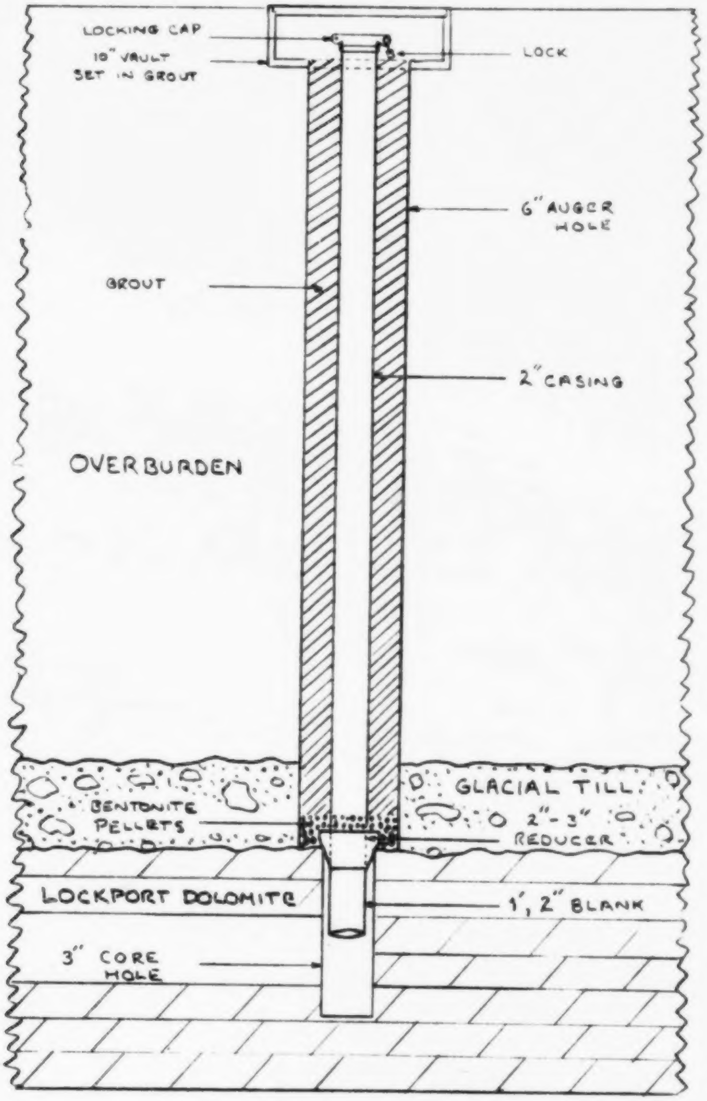
LOCKING CAP
10" VAULT SET IN GROUT
LOCK
6" AUGER HOLE
GROUT
2" CASING
OVERBURDEN
GLACIAL TILL
BENTONITE PELLETS
2"-3" REDUCER
LOCKPORT DOLOMITE
1', 2" BLANK
3" CORE HOLE

FIGURE 3-5.

TYPICAL WELL TOP OF DOLOMITE

15. Insure site is clear of all equipment and drums and that concrete culverts are installed properly before leaving site.

16. Place lock onto well cap and proceed to decontamination, following the procedure in Section 5 of this document.

D. DEEP BEDROCK WELL DRILLING PROCEDURE

3.10 PERSONNEL

The requirements for personnel are as follows:

- 1 supervisory geologist to oversee drilling operations and log progress of well
- 1 driller to conduct drilling operation
- 1 driller's helper to assist in the conduct of drilling operations
- 1 QC/QA Officer to conduct periodic inspection
- 1 safety officer to conduct periodic inspections.

3.11 EQUIPMENT

Equipment requirements are as follows:

- 1 drilling rig with all associated equipment
- required safety equipment (see Safety Manual)
- 1 conductivity meter
- 1 fiberglass measuring tape - weighted at the end
- 1 water soluble marking pen
- clipboard, well log form, pens, pencils
- 1 field notebook
- 1 watch
- 1 thermometer (C-scale).

3.12 PROCEDURE

1. Set up to drill over the wooden stake placed on the site by the survey crew.

   STOP - No drilling is to commence until a Work Authorization form is issued, and all personnel are wearing their safety equipment required in the Health and Safety Plan.

2. Begin to auger with a 6-inch diameter auger bit into the overburden.

3. As the drilling progresses, take samples of the overburden material each time it appears a change has taken place in the physical composition of the drilled material. Record all descriptions of the overburden material onto the well log form (see Figure 3-3). NOTE: Be extra sensitive to any changes in the moisture content of the material as it is brought up by the auger bit. All cuttings and disposable safety equipment are to be disposed of in 55 gallon drums with lids until the area is declared clean.

4. To verify the contact with bedrock, remove the center of the auger, replace with a split spoon and proceed to hit the rock until refusal is certain.

5. Once hard rock is verified, pull auger back 1 foot and install clay pellets.

6. Remove all remaining auger flights and drive 4-inch drain casing firmly into bedrock.

7. Using a 3-1/2 inch tri-cone bit, wash the 4-inch casing clean of all material collected during the setting process. NOTE: All wash material being removed from the hole must be captured in a trough and pumped into a tanker truck for safe disposal.

8. Grout the 4-inch casing in the hole, allowing the grout to back-fill into the casing to ensure a firm seal.

9. Allow this to set overnight for hardening of the grout.

10. Based upon the instructions of the work authorization form, core with a 3-inch NX core barrel through the Lockport Dolomite until contact is verified with the Rochester Shale. Careful measurement of drilling water used should be made, to enable an accurate determination of water bearing zones. In addition, conductivity and temperature readings should be taken at each 1-foot interval.

11. Once the Rochester Shale has been reached, remove the core bit and take a gamma-ray log of the entire section.

12. Upon completion of the gamma-ray logging, back fill the hole with grout to within 5 feet of the overburden bedrock interface, and complete the well as shown in Figure 3-6. NOTE: Take a final water level reading prior to grouting of the hole.

13. Once the well is installed, take final static water level prior to leaving the site.

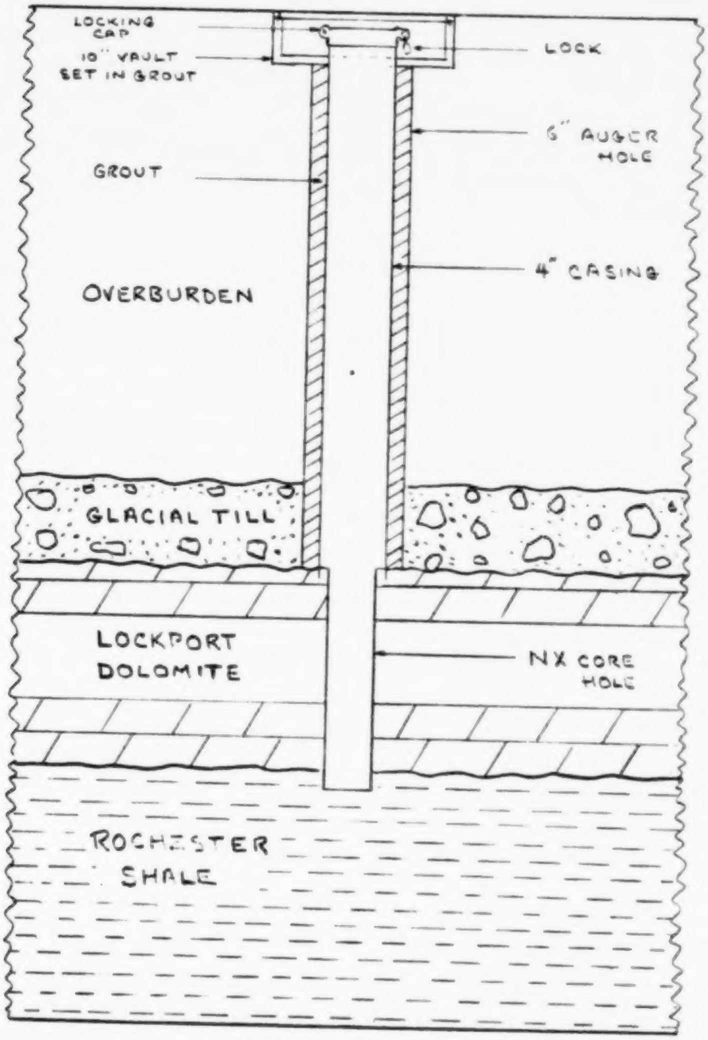
LOCKING CAP
10" VAULT SET IN GROUT
LOCK
6" AUGER HOLE
GROUT
4" CASING
OVERBURDEN
GLACIAL TILL
LOCKPORT DOLOMITE
NX CORE HOLE
ROCHESTER SHALE

FIGURE 3-6.

TYPICAL WELL TOP OF SHALE

14. Insure site is clear of all equipment and drums and that concrete culverts are installed properly before leaving site.

15. Place lock onto well cap and proceed to decontamination, following the procedure in Section 5 of this document.

E. DECONTAMINATION PROCEDURES

The decontamination protocol is designed to minimize health and safety hazards and prevent interwell contamination.

3.13 PERSONNEL

Decontamination is carried out by drilling personnel under the direction of the supervisor geologist with periodic inspection by the onsite JRB Health and Safety Officer and QA/QC Officer.

3.14 EQUIPMENT

This protocol is applicable to all contaminated equipment, tools, and clothing. Decontamination is carried out at the designated cleanup area using a high pressure water spray, a brush, and a shovel. Personnel are required to wear rubber boots, gloves, protective coveralls, goggles, and hard hats. Residual materials are deposited in a cuttings barrel.

3.15 PROCEDURE

All contaminated items are transported to the designated cleanup area for decontamination at the end of each working day and/or prior to moving between sites.

Upon arrival at the cleanup area, position equipment so that it can be sprayed down with clear access to the central drain for runoff. If necessary spray off a convenient portion of the cleanup area to place decontaminated items while draining. Remove and lay out on the spray pad all movable items. Scrape or knock off readily removable bulk wastes and gather together in a dry side area with a shovel. If applicable, spray off transport vehicle first, using a brush if necessary to remove all visible waste.

Then spray off all smaller items, using a brush if necessary to remove visible waste. Continuously gather any accumulating solid wastes and add to bulk waste pile with a shovel. Move all decontaminated items to the clean drainage area. Continue process until all items have been treated. Pay particular attention to items having recessed areas or hollow centers such as pipes or auger flights.

When all items have been processed inspect and repeat cleanup procedure if necessary. When items are clean, replace on drill rig or in transport vehicle. Shovel all bulk wastes into the cuttings barrel marked with the site number. Spray down cleanup area. Remove disposable equipment or clothing items (respirator cartridges, coveralls, etc.), and place in cuttings barrel. Seal barrel and place in designated pickup area. Depart for next assignment.

F. PUMP TEST PROCEDURE

(Yet to be determined)

G. AQUIFER TESTS

(Yet to be determined)

4.0 SAMPLING PROTOCOL

GROUNDWATER SAMPLES IN THE LOVE CANAL AREA

This plan has been designed for the purposes of sampling the groundwaters in the vicinity of Love Canal. The samples will be taken from 2-inch diameter monitoring wells. The sampling plan, outlined below, is the step-by-step process that will be followed by JRB personnel in completing this task.

4.1 PERSONNEL

The requirements for personnel are as follows:

- 2 environmental chemists to perform the sampling
- 1 safety and health expert to oversee the quality control procedures
- 1 senior geologist to inform the sampling crew of any information that has been previously gained at that well
- All personnel will be wearing respirators, safety glasses, disposable coveralls, boots, hard hats, and neoprene gloves.

4.2 PURGING THE WELL

Prior to sampling, the well will be pumped to ensure that a representative "clean" sample is obtained during the sampling process. The type of technique employed at each well will be pumping by means of a centrifugal pump.

- Centrifugal pump equipped with a 3/4-inch black steel pipe will be inserted in the well to midway in water zone
- Pipes will be cleaned prior to insertion in the well by means of the standard cleaning procedures*
- Any water collected for measuring conductivity, pH, or cleaning water will be deposited in the drum with the pump water
- Water will be pumped from the well in an amount equal to 10 times the volume of water in the well, or until the conductivity of the water coming out stabilizes

---

*Standard cleaning procedures: soap and water followed by deionized water rinse.

- Pumped water will be collected in 55-gallon drums, then taken to a tank truck to be pumped out
- 5 gallons of fresh water will be pumped through the pumps at the end of each day to prevent transport of contaminated material and cut down on the wear and tear of the pumps
- If the well should be pumped dry during the pumping process, the well will be allowed to stand for 24 hours or until it fills with water before bailing begins.

4.3 BAILING

After the well has been pumped, the bailing for the actual sample collection will be done. The bailing activity requires the following steps:

- Wash teflon bailer using standard cleaning procedures
- Lower bailer into well with hand-operated winch and stainless steel cable
- Allow water to fill from bottom of bailer
- The bailer, when full, is then raised from the well, using the winch
- The water in the bailer is then poured into the sample container all the way to the top of bottle. Proper preservative is then added to the sample container. (See preservative discussion in the next section.)
- Sample container is then sealed and labeled according to the GCA chain of custody procedures
- The chain of custody forms are then filled out
- This process is repeated for as many samples that are to be taken at each well
- Bailers are stored in plastic bags when moving between sites
- The samples are then put in thermal crates with ice and taken to the GCA sample bank.

4.4 SAMPLING PROCEDURES

All sample vessels will be prepared by GCA and provided to JRB cleaned by the proper cleaning techniques, referenced as Attachment I.

4.4.1 Sampling for Volatile Organics

- The groundwater sample is to be collected in two 40 ml vials with Teflon-faced silicone septa and Teflon-lined screw caps and maintained at 4°C until the sampler's responsibility has been relieved at the Sample Bank.

- Bailer Preparation

  1. Wash with Alconox and tapwater followed by a distilled Deionized rinse.

- Sample Collection Procedures

  1. When sampling for volatile organics, the method used shall not cause stripping of the volatiles from the water column. The stripping will occur when there is turbulence in the sample water or a reduction in pressure.
  2. If a Teflon bailer or other appropriate device is used to collect or purge the sample water, it must be precleaned according to procedures in 7.2.1, B of the GCA Quality Assurance Plan.
  3. The collection of the groundwater sample is to ensue after purging the well. The sample shall be collected using a Teflon or stainless steel bailer; the Teflon is preferred.
  4. The bailer is to be lowered toward the bottom of the casing and allowed to fill. To lower and raise the bailer, use stainless steel wire. Raise the filled bailer slowly out of the water so a minimum of disturbance will result as the sample is retrieved.
  5. The sample water in the bailer shall be poured carefully into a tilted 40 ml sample vial so the sample stream moves gently down the side of the vial with minimal turbulence.
  6. Continue pouring the sample water from the bailer as described in Step 7 until the vial is filled and overflows for a few seconds.
  7. Place the Teflon-coated silicone carefully into place, and screw on the Teflon-lined cap firmly.
  8. Invert the vial to check for air bubbles. If any are present, select another vial and resample. Discard the vial with entrapped air.
  9. Place the sample bottle in an appropriate container to be maintained at 4°C throughout the sampling and transportation period.
  10. The samples are to be transported and transferred to the GCA Sample Bank within four (4) hours of collection, strictly observing the Chain-of-Custody procedures described in Section 5 of GCA Quality Assurance Plan.

4.4.2 Sampling for Semivolatile Organics

- The groundwater sample is to be collected in a one (1) gallon amber glass bottle with a Teflon-lined screw cap and maintained at 4°C until the sampler's responsibility has been relieved at the Sample Bank.

- Sample Collection Procedures

    1. The sampling will be done according to Sampling for Organic Chemicals and Micro-organisms in the Subsurface (EPA-600/2-77-176), using Section 4, page 5 - Sampling Procedures, Preliminary Operations, and Acquisition of Grab Samples.
    2. Samples will be collected by bailing - see 1.4.1 for a description.
    3. Place the sample bottle, after labeling, into an appropriate container maintained at 4°C throughout the sampling and transportation period.
    4. Samples are to be transported and transferred to the GCA Sample Bank within four (4) hours of collection, strictly observing the Chain-of-Custody procedures described in Section 5 of GCA Quality Assurance Plan.

4.4.3 Sampling for Pesticides

- The groundwater sample is to be collected in a one (1) gallon amber glass bottle with a Teflon-lined screw cap and maintained at 4°C until the sampler's responsibility has been relieved at the Sample Bank.
- Sample Collection Procedures

    1. The sampling will be done according to Sampling for Organic Chemicals and Micro-organisms in the Subsurface (EPA-600/2-77-176), using Section 4, page 5 - Sampling Procedures, Preliminary Operations, and Acquisition of Grab Samples.
    2. Samples will be collected by bailing - see 1.4.1 for a description.
    3. Place the sample bottle, after labeling, into an appropriate container maintained at 4°C throughout the sampling and transportation period.
    4. Samples are to be transported and transferred to the GCA Sample Bank within four (4) hours of collection, strictly observing the Chain-of-Custody procedures described in Section 5 of GCA Quality Assurance Plan.

4.4.4 Sampling for TCDD

- The groundwater sample is to be collected in a one (1) gallon amber glass bottle with a Teflon-lined screw cap and maintained at 4°C until the sampler's responsibility has been relieved at the Sample Bank. TCDD samples will be collected on a "requested" basis and will not be part of the regular sampling protocol.

- Sample Collection Procedures

  1. The sampling will be done according to Sampling for Organic Chemicals and Micro-organisms in the Subsurface (EPA-600/2-77-176), using Section 4, page 5 - Sampling Procedures, Preliminary Operations, and Acquisition of Grab Samples.
  2. Samples will be collected by bailing - see 1.4.1 for a description of the bailing technique.
  3. Place the sample bottle, after labeling, into an appropriate container maintained at 4°C throughout the sampling and transportation period.
  4. Samples are to be transported and transferred to the GCA Sample Bank within four (4) hours of collection, strictly observing the Chain-of-Custody procedures described in Section 5 of GCA Quality Assurance Plan.

4.4.5 Sampling for Inorganics

- The groundwater sample is to be collected in (125 ml) glass with a Teflon-lined screw cap and maintained at 4°C until the sampler's responsibility has been relieved at the Sample Bank.
- Sample Collection Procedures

  1. The sampling will be done according to Sampling for Organic Chemicals and Micro-organisms in the Subsurface (EPA-600/2-77-176), using Section 4, page 5 - Sampling Procedures, Preliminary Operations, and Acquisition of Grab Samples.
  2. Samples will be collected using Teflon bailers following the format outlined in 1.4.1.
  3. Place the sample bottle, after labeling, into an appropriate container maintained at 4°C throughout the sampling and transportation period.
  4. Samples are to be transported and transferred to the GCA Sample Bank within four (4) hours of collection, strictly observing the Chain-of-Custody procedures described in Section 5 of GCA Quality Assurance Plan.

4.4.6 Sampling for Total Organic Carbon (TOC)

- The groundwater sample is to be collected in 500 ml clear glass jars with Teflon-lined screw caps and maintained at 4°C until the sampler's responsibility has been relieved at the Sample Bank.

- Sample Collection Procedures

  1. When sampling for TOC, the method used shall not cause stripping of the volatiles from the water column. The stripping will occur when there is turbulence in the sample water or a reduction in pressure.
  2. Samples will be collected by bailing - see 1.4.1 for a description.
  3. Samples will be collected in 500 ml Teflon-lined clear glass jars.
  4. Place the sample bottle, into an appropriate container maintained at 4°C throughout the sampling and transportation period.
  5. Samples are to be transported and transferred to the GCA Sample Bank within four (4) hours of collection, strictly observing the Chain-of-Custody procedures described in Section 5 of GCA Quality Assurance Plan.

4.4.7 Sampling for Total Organic Halogens (TOX)

- The groundwater sample is to be collected in 500 ml clear glass jars with Teflon-lined screw caps and maintained at 4°C until the sampler's responsibility has been relieved at the Sample Bank.
- Sample Collection Procedures

  1. When sampling for TOX, the method used shall not cause stripping of the volatiles from the water column. The stripping will occur when there is turbulence in the sample water or a reduction in pressure.
  2. Samples will be collected in Teflon bailers following the procedures outlined in section 1.4.1.
  3. Place the sample bottle in an appropriate container maintained at 4°C throughout the sampling and transportation period.
  4. Samples are to be transported and transferred to the GCA Sample Bank within four (4) hours of collection, strictly observing the Chain-of-Custody procedures described in Section 5 of GCA Quality Assurance Plan.

4.5 QUALITY ASSURANCE/QUALITY CONTROL DURING SAMPLING

To assure that the sampling procedures are not causing any contamination of the samples, JRB plans to use a QA/QC process to verify cleanliness. At 5% of the wells QA/QC will be performed by pouring dionized water over the sampling equipment and collected for TOC and TOX analysis. If the amount of TOC and TOX of the rinse water is greater than (% error of the analysis) to the deionized water, that sample well will be resampled and the samples taken will be invalidated.

Field blanks will be included with each day's samples according to the GCA Protocol referenced as Attachment II.

SAMPLE SCHEDULE FOR DETAILED ANALYSIS*/**

WEEK of 9/21/80

Monday - 21

Tuesday - 21

Wednesday - 21

WEEK of 9/28/80

Monday - 22

Tuesday - 22

Wednesday - 22

WEEK of 10/5/80

Monday - 21

Tuesday - 21

Wednesday - 21

WEEK of 10/12/80

Monday - 21

Tuesday - 21

Wednesday - 21

The sampling schedule is dependent upon the drilling of 12 wells per day and that those wells are producing a sufficient volume of water. Furthermore, this schedule will not be met if we do not get sample bottles and preservatives.

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*Includes 5% QA/AC check

**Does not include TOC/TOX Analysis

SAMPLE SCHEDULE FOR TOC/TOX

WEEK of 9/7/80	20
WEEK of 9/14/80	44
WEEK of 9/21/80	44
WEEK of 9/28/80	44
WEEK of 10/5/80	44
WEEK of 10/12/80	44

Samples will be taken on Thursday and Friday, most will be on Friday.

---

*Includes 5% for QA/QC analysis.

ATTACHMENT I

TO: GTI, AES, JRB, GCA Field Personnel
FROM: Rose Mary Ellersick
SUBJECT: Cleaning Procedures for Sample Containers - LC1-619-026
DATE: September 11, 1980

All sample containers, covers, and liners are to be washed with an Alconox detergent solution and thoroughly rinsed with tap water; this detergent wash may be accomplished in a commercial dishwasher. Glass bottles may be dried in an oven, or air-dried; solvent rinsed bottles should be air-dried to avoid contaminating the oven with solvents. Specific cleaning required is listed below according to the media sampled and the type of sample collected.

Air

Volatile Organics - EPA is providing clean containers

Semivolatile Organics, Pesticides, TCDD - The aluminum foil used to wrap the collected sample must be rinsed with hexane and air-dried before use.

Glass containers - Alconox wash, thorough tap water rinse, thorough drying, thorough hexane rinse, thorough air-drying.

Water and Soil

Volatile Organics - Glass containers - Alconox wash, thorough tap-water rinse, thorough DDI water rinse, oven-drying at 105°C for one hour.

Semivolatile Organics, Pesticides, TCDD - Glass containers - Alconox wash, thorough tap-water rinse, thorough DDI water rinse, thorough Drying, thorough methylene chloride rinse, thorough air-drying.

Inorganic-Metals - Glass containers - Alconox wash, thorough tap-water rinse, thorough nitric acid rinse*, thorough DDI water rinse. thorough drying.

* Concentrated Ultrex Acid (or highest quality available) should be mixed with equal amounts of DDI water to provide the rinse solution.

Inorganic - Flouride, Nitrate - Plastic Containers - Alconox wash, thorough tap-water rinse, thorough DDI water rinse, thorough drying.

Radioactivity - EPA is providing clean containers.

The cleaned sample containers, covers, and liners are to be considered as sampling equipment. They should be stored in a contamination-free atmosphere and measures taken to ensure that no tampering has occurred. Appropriate measures include the use of evidence tape or custody seals on boxes on cleaned containers, or doors of storage rooms.
The GCA Sample Bank will provide this tape.

Cleaning procedures have been provided by GCA in Appendix A: Sampling Procedures. They are essentially the same as those given here; when a discrepancy is found, this memo should be followed. All personnel cleaning sample containers or other equipment should be working from written instructions which agree with this memo.

Rose Mary Ellersick
RoseMary Ellersick
QA Manager

cc: R. Bradway
D. Cogley
K. McGregor

ATTACHMENT II

TO: GTI, JRB, AES, GCA Field Personnel
FROM: RoseMary Ellersick
SUBJECT: Clarification of Field Blanks
DATE: September 11, 1980

The purpose of a field blank is to monitor sample handling procedures, including container cleanup, and document that contamination has not occurred. The field blank is to be carried to the sampling site, not exposed to the sampled air, water, soil, etc. but handled in the same way as collected samples. The field blank will remain in the refrigerated container (picnic cooler, etc.) used to transport collected samples while the sampling team collects its assigned samples.

The field blank is analyzed by the laboratory analyzing the samples, therefore, a field blank is required for each type of sample collected and for each media (air, water, soil, etc.) sampled. The following table should clarify this. Unless otherwise stated, the field blank is DDI water in a clean sample container of the same type and size used for collected samples.

If the same tean is sampling different types of waters, one field blank/sample type will be sufficient. If the same team is sampling different media, such as water and soil or water and sediment, or soil and sediment, field blanks for each sample type must be provided for each media.

FIELD BLANKS REQUIRED PER SAMPLING TEAM PER SAMPLING DAY

Air

Sample Type	Field Blank
Volatiles	Tenax Cartridge
Semivolatiles/Pesticides	Foam Plug Module

Water, Soil and Sediment

Sample Type
Volatiles
Semivolatiles
Pesticides
Nitrates, Flourides
Metals
TCDD
TOC
Total Halogenerated Organics (THO or TOX)

Rose Mary Ellersick
RoseMary Ellersick
QA Manager

cc: R. Bradway
K. McGregor
D. Cogley

5.0 DOCUMENT CONTROL PROCEDURES

5.1 GENERAL INFORMATION

The purpose of document control is to ensure that all project documents will be accounted for when the project is complete. Table 5-1 lists the principal items subject to document control with records maintained by the person listed as Document Control Officer (DCO) for that document. Table 5-2 gives a more complete listing of documents generated during the project. GCA will provide a DCO onsite at the Sample Bank who will maintain overall document control for all items passing through the Sample Bank.

Each subcontractor shall identify a DCO for its operations who will be responsible for issuing, controlling and maintaining records of controlled documents. At the conclusion of subcontract activity, all controlled documents with records shall be provided to GCA for inclusion in an overall project document inventory.

Procedures described in Section IX of "Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors," Environmental Protection Agency, National Enforcement Investigations Center, Denver, Colorado, April 1980, shall be followed.

5.2 QA PROJECT PLAN

Each page will contain a heading on the upper right-hand corner that indicates Section Number, Revision Number, and Page Number of Total Pages in the section. The appropriate document numbering for subcontractor QA Plans is included with subcontract transmittal letter. When a revision to a QA Plan is made, the Table of Contents will be retyped to show the most current revision of each section, and both the revision and the revised Table of Contents will be distributed to all recipients of the original QA Plan. These recipients will be listed at the end of the QA Plan Table of contents and in the QA Manager's records.

5.3 PROJECT NUMBERING SYSTEM

5.3.1 Project Code

The project code for the Love Canal Study is LC 1-619-026; this number must appear on sample identification tags, custody records, logbooks, data sheets, control charts, project memos and reports, document control logs, corrective action forms and logs, QA Plans, and all other project records.

TABLE 5-1.

DOCUMENT CONTROL REQUIREMENTS

Item	Issued by	Numbering	Interim responsibility	DCO
Sample Collection Tags	Sample Bank	Preserialized	Sampling Subcontractor	GCA Sample Bank
Custody Records	Sample Bank	Preserialized	See Section 5.0	See Section 5.0
Field Logbooks	Sample Bank	LC 1-619-026-	Sampling Subcontractor	Sampling Subcontractor
Field Data Sheets	Sample Bank	LC 1-619-026-	Sampling Subcontractor	GCA Sample Bank
Written Communication	Various People	Number and page of personal notebook where item is cited	Recipient	Subcontractor
Photos or Drawings	Originator		Originator	Subcontractor
Analytical Sample Tags	Sample Bank	Preserialized	Shipper	Analytical Subcontractor
Laboratory Notebooks	Subcontractor	LC 1-619-026-	Analytical Subcontractor	Analytical Subcontractor
Analytical Data Sheets	GCA	LC 1-619-026-	Analytical Subcontractor	Analytical Subcontractor
QA Project Plan	GCA	Section, Revision, Page Number of Total Pages	GCA QA Manager	GCA QA Manager
QA Project Plans	Subcontractors		Subcontractors	

TABLE 5-2. SUGGESTED DOCUMENT CODES

Document	Code Letter
Project Work Plans	A
Project Logbooks	B
Sampling Logbooks	C
Sampling Data Sheets	D1, D2, etc.
Sampling Coding Form	E
Laboratory Notebooks	G
Laboratory Data Sheets	H1, H2, etc.
Laboratory Coding Form	J1, J2, etc.
Sample Logs	L1, L2, etc.
Internal Memos	M
External Written Communication	N
Confidential Information	O
Photos, Maps, Drawings	P
QA Plan	Q
Reports	R
Final Report	FR
Miscellaneous	X

5.3.2 Preserialized Documents

Sample collection and analytical tags, and custody records have preprinted serial numbers on them. It is not necessary that a tag number match a custody record number; it is necessary that each subcontractor DCO maintain records which account for all serially numbered items received. If tags or custody records are damaged, lost or destroyed before use for their intended purpose, the serial number of the item and its disposition must be recorded.

5.3.3 Other Documents

Sample identification tags and custody records are accounted for by their preserialized numbers. Other documents use the following numbering scheme to enable unique identification of each item:

Project Code	Subcontractor Code	Document Code	Serial Number
LC 1-619-026-	-00-	-A-	-00001

Table 4-2 presents a suggested document code. Each subcontractor's DCO will finalize the system to be used for their documents; it is desirable that consistent document codes be used by all subcontractors to facilitate compiling the overall project document inventory.

6.0 CHAIN-OF-CUSTODY PROCEDURES

6.1 GENERAL INFORMATION

The procedures identified here have been established for this project by the GCA QA Manager working with NEIC staff members and following guidelines as developed from the following two publications:

- NEIC Policies and Procedures Manual, EPA-330/9-78-001R, Section II
- Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, NEIC, April 1980, Sections VIII and IX.

The purpose of chain-of-custody procedures is to document the identity of the sample, and its handling, from its first existence as a sample until its ultimate disposal. It must be possible, via a custody record, to trace a sample through all transfers of custody and to verify that it is the same sample collected.

In order to establish that a sample is valid, it is also necessary to document that no tampering has occurred - either to the sample itself, the sampling equipment used or the environment sampled. This is done by the use of evidence tape, locks and custody seals, and documented entries noting their condition in field and laboratory log books. The custody record must also document any tampering that occurred; the absence of any such comments indicates no tampering during the period of custody.

The GCA Sample Bank will provide and dispense evidence tape, sample tags, custody records, logbooks, field and laboratory data sheets, custody seals and locks as appropriate. Custody procedures are presented here in considerable detail so that they may be uniformly followed.

The sampling subcontractor shall identify an onsite person responsible for ensuring that custody procedures are followed and for referring any tampering cases or other questions ot GCA's onsite QA coordinator who is responsible for onsite custody procedures. Analytical subcontractors shall also identify a person responsible for custody matters.

6.2 [NOT APPLICABLE]

6.3 SAMPLE IDENTIFICATION

Sample collection tags have been designed for air, water, soil and sediment, and biota or other samples and are shown in Figures 6-1 and 6-2. They are a different color for each media and are serially numbered starting with different blocks of numbers for each media. The tags are preprinted to ensure that the required information is provided on each tag. Each collected sample, including duplicates and field blanks, shall have a completely filled-in sample tag securely attached to it. Duplicates and field blanks shall be so identified in the "Remarks" section of the tag.

The person who physically collects the sample is the SAMPLER and signs the sample tag. He may presign the tag if he will be in hazardous conditions when sampling. Another member of the team may enter the exact time of sample collection; all team members present shall be recorded in the logbook. The SAMPLER initiates the custody record for transfer of samples to the Sample Bank.

After the collected samples have been transferred to the Sample Bank and documented there, they will be renumbered and identified with a new sample analysis tag for shipment to analytical laboratories. This renumbering step enables the submittal of duplicate samples and QC samples as blinds to the analytical laboratory. The same analysis tag (Figure 6-3) is used for all samples; it is a different color and numbered with a block of numbers different from other tags.

An important aspect of sample identification traceability is the system of cross-indexing logbooks maintained at the Sample Bank and the computer file which can correlate any one of the identifying numbers with a specific sample. These numbers include those found on the:

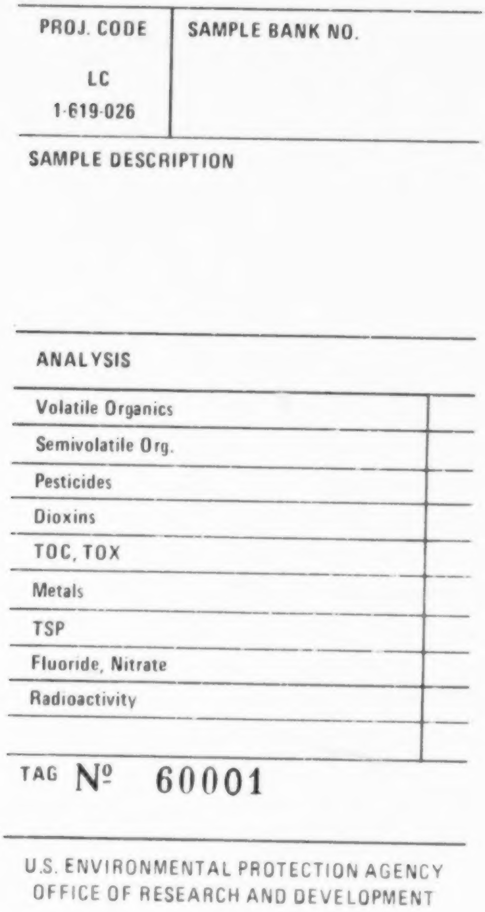
- Sample Collection Tag
- Custody Record transferring custody to the Sample Bank
- Sample Bank Master Log
- Analytical Sample Tag
- Custody Record transferring custody to the shipper and then to the Analytical Laboratory.

6.4 SAMPLE TRANSFER PROCEDURES/CHAIN-OF-CUSTODY RECORDS

There are many transfers of custody in this program since materials are shipped to the Sample Bank for distribution to the sampling team or inclusion with collected samples, and shipped from the Sample Bank to analytical laboratories. The collection medium used for air sampling becomes part of the sample itself after collection; therefore, the laboratory preparing Tenax cartridges or polyurethane foam plugs must initiate a custody record and include it with their shipment of Tenax or foam. Laboratories providing QC samples must also initiate a custody record.

6.4.1 Chain-of-Custody Record Sheets

Customized record sheets, following the NEIC format, have been provided for this program and are shown as pages 8 through 13 of this section. Pages 8 and 9 are used for shipping collection media and QC samples to the Sample Bank; pages 10 through 12 are used for samples collected in the field; and



PROJ. CODE LC 1-619-026	SAMPLE BANK NO.

SAMPLE DESCRIPTION

ANALYSIS

Volatile Organics	
Semivolatile Org.	
Pesticides	
Dioxins	
TOC, TOX	
Metals	
TSP	
Fluoride, Nitrate	
Radioactivity	

TAG № 60001

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT

FIGURE 6-3.

ANALYSIS TAG

Figure 6-4 is used for all shipping of samples to analytical laboratories. They are two-part carbonless copy forms which correlate with the sample identification tags; requested information has the same heading on both.

The custody records are used for a packaged lot of samples; more than one sample will usually be recorded on one form. More than one custody record sheet may be used for one package, if necessary. Their purpose is to document the transfer of a group of samples traveling together; when the group of samples changes, a new custody record is initiated. The original of the custody record always travels with the samples; the initiator of the record keeps the copy. When custody of the same group of samples changes hands several times, some people will not have a copy of the custody record. This is acceptable as long as the original custody record shows that each person who had received custody has properly relinquished it.

6.4.2 Using the Custody Record Sheet

- The originator fills in all requested information from the sample tags (except in the case of air collection media and external QC samples).
- The person receiving custody checks the sample tag information against the custody record. He also checks sample condition and notes anything unusual under "Remarks" on the custody form.
- The originator signs in the top left "Relinquished by" box and keeps the copy.
- The person receiving custody signs in the adjacent "Received by" box and keeps the original.
- The Date/Time will be the same for both signatures since custody must be transferred to another person.
- When custody is transferred to the Sample Bank or an analytical laboratory, blank signature spaces may be left and the customized last "Received by" signature box used. Another approach is to run a line through the unused signature boxes.
- In all cases, it must be readily seen that the same person receiving custody has relinquished it to the next custodian.

6.4.3 Chain-of-Custody Forms Used for a Volatile Organic Air Sample

An air sample is used for example because it requires several different custody forms.

Collection Media

- Initiated by Tenax preparing lab which keeps copy
- Custody transferred to Sample Bank
- Custody transferred to subcontractor's sampling team member

ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development

CHAIN OF CUSTODY RECORD

Project Code LC	Project Name LOVE CANAL STUDY (1-619-026)							ANALYSIS										REMARKS
TO:	SAMPLE DESCRIPTION																	
Sample Bank No.	Tenax	Polyfoam	Hi-Vol	Water	Soil	Sediment	Other	Volatile Organics	Semivolatile Organics	Pesticides	Dioxins	TOC, TOX	Metals	TSP	Fluoride, Nitrate	Radioactivity		

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks	

№ 2000 Distribution: Original Accompanies Shipment; Copy to Sample Bank Files

FIGURE 6-4.

If the whole lot received by the Sample Bank is transferred to the sampling team, one custody record is used throughout; the sampling team keeps the original. If only part of the original received lot is transferred, a new Collection Media Custody form is initiated by the Sample Bank which keeps the copy. The sampling team keeps the original.

Collected Air Samples

- Initiated by sampling team member who keeps copy
- Custody transferred to Sample Bank which keeps original and maintains internal custody procedures
- Samples recorded in Master Log, QC samples included, and Sample Bank numbers assigned. New sample tags attached.

Analytical Samples

- Initiated by Sample Bank
- Custody transferred to shipper, such as Federal Express, before packing the original of custody form in with the samples
- Shipper does not keep copy of custody form. Sample Bank keeps it and a copy of the bill of lading
- Custody transferred to analytical lab which keeps original record
- Analytical lab maintains its internal custody procedures.

6.4.4 Custody Seals

Custody seals are narrow strips of adhesive paper used to demonstrate that no tampering has occurred. They may be used on sampling equipment or a house door, but they are intended for use on a sample transport container which is not secured by a padlock. They are not intended for use on individual sample containers.

6.4.5 Custody of Shipped Samples

When a group of samples with its custody form is to be shipped, the shipper (e.g., Federal Express) must sign the custody record before the package is closed so that he can verify the number and identification of the samples if he so desires. The shipper is not required to do so.

The package is then closed with a strong, keyed padlock or strapping tape and custody seals.

NEIC's recommended procedure for custody seal use on shipping boxes is as follows:

- Place paper side of seal against box and wrap strapping tape across the middle of the seal and at least twice around the box
- Cut the tape so that the ends overlap at the seal
- Fold the seal to itself over this overlap and sign the custody seal
- Repeat this procedure as many times as necessary to seal the box for shipment.

The person receiving custody of shipped samples must document the condition of the locked, or strapped and sealed shipping box on arrival. It must be checked that neither the tape or the custody seals has been cut or otherwise tampered with. If the paper seal has been damaged in shipping but it is clear that the shipping box has not been opened, further handling of the samples may proceed. If tampering is suspected, the designated Sample Custodian shall notify the GCA Sample Bank.

6.5 LABORATORY CUSTODY PROCEDURES

The Sample Bank, the sampling subcontractor's laboratory area, and each subcontracting analytical laboratory are considered to be working laboratories. Each laboratory shall have a designated Sample Custodian who implements a system to maintain control of samples. It is assumed that the laboratory is not open to the general public but has access restricted to employees known to each other. Each handling of the sample to renumber, subdivide, preserve, etc., does not have to be documented in this situation. Each subcontractor shall describe his laboratory custody procedures in his QA Plan.

Appendix Q
Plan 5.2

Empire Soils Investigations, Inc.

QA Plan

LC 1-619-026
Page 1 of 1

Document Control Officer (DCO) - Brian Schleifer, QA/OC/SHO

I. SITE CLEARANCE
   A. To be provided by others
      1. Power
      2. Gas
      3. Phone
      4. Water
      5. Sewer
      6. Other (to be specified)
   B. Work Authorization
      1. Documented
      2. Delivered to DCO-Empire Soils Investigations, Inc.
   C. Site Sketch
      1. Document all surrounding fixed objects, i.e., catch basins, telephone poles, swales, etc.
      2. Address

II. MOBILIZATION
   A. Equip drilling rig
      1. Augers
      2. Casing
      3. Half-barrel
      4. Hose
      5. Barrels for cuttings
      6. General drilling tools
   B. Deliver Tanker
      1. Transport "contaminated" drilling water
      2. Storage for "contaminated" drilling water
   C. Secure Site
      1. Visable barrier, i.e., twine and lath fence with florescent flagging per JRB Associates, Inc.
      2. Safety equipment for drillers

EMPIRE SOILS INVESTIGATIONS, INC.

Task I: Determination of Dolomite Stratigraphy in the Love Canal Area

Empire Soils Investigations, Inc., will drill wells through the Lockport Dolomite, in areas of Niagara County, believed to be free of contamination. The Supervisory Geologists will determine the locations of these wells with the concurrence of GCA.

All equipment and materials to be used during the drilling activities and well installation or plugging of wells, will be carefully cleaned by washing, with approved cleansing materials and rinsed with organic free water, per specifications.

The overburden drilling will be done using 6-inch hollow stem augers. The cuttings from this operation are to be put into barrels, which will be labelled. Six-inch casing will be installed down to the bedrock. This casing will be washed out, using a 5-5/8-inch roller bit and City water from the nearest fire hydrant. A 4-inch welded steel casing will then be seated and grouted into the top of rock. As the grout is placed outside of the 4-inch casing, the 6-inch casing shall be removed. The grout will require at least 24-hours to set-up, prior to the coring of the dolomite.

After resetting the drill rig on the site, "NX" core samples for the entire length of the bedrock section shall be provided to the Prime Contractor. The core samples will be placed in core boxes provided by Empire Soils Investigations, and labelled by the Supervisory Geologist.

Once the Supervisory Geologists have determined, the location(s) of the water-bearing zone(s) Empire Soils Investigations, Inc., will perform Packer Tests, per Supervisory Geologists direction. Upon completion, the wells will be plugged per specifications by New York State, when notified in writing by GCA.

All water used for drilling and testing purposes will be pumped into tankers. This water will be stored along with cuttings, until written direction has been received as to where disposal of each may take place.

Task II: Drilling of Initial Test Wells

Empire Soils Investigations, Inc., will drill ten (10) test wells to the top of the glacial till. Six-inch hollow stem augers will be used to produce an open hole in the overburden. A 2-inch stainless steel screen will be welded to a 2-inch steel blank pipe and installed to the bottom of the hole. The hole will be backfilled with washed sand to a depth determined by the Supervisory Geologist. The hole will be backfilled with grout from that depth to within three feet of the existing grade. A protective casing will then be installed.

Empire Soils Investigations, Inc., will drill ten (10) test wells into the upper part of the underlying dolomite. Six-inch hollow stem augers will be used to produce an open hole down to the top of the bedrock. Six-inch casing will be installed down to the top of rock to prevent migration of water from one zone to another. This casing will be washed out and a hole will be drilled into the dolomite, using a roller-rock bit. A 2-inch well point will be seated into the bedrock. Bentonite pellets will be placed at the top of rock to develop a seal between the overburden and the bedrock. The 2-inch pipe will then be grouted in-place. A protective casing will then be installed over the well.

All cuttings will be stored in barrels and all water used for drilling will be stored in tankers until written notification has been submitted to DCO - Empire Soils Investigations, Inc. At this time proper disposal will take place.

Task III: Drilling Monitoring Wells

Empire Soils Investigations, Inc., will drill and geologically log 110 pair of wells. The pair will consist of a well drilled to the top of the underlying glacial till and a well drilled into the upper part of the underlying dolomite.

As of this writing the following procedure has been accepted by GCA in concurrance with the Supervisory Geologists. The intial installations were typical to description of installation of wells in Task II.

The wells into the top part of the dolomite are to be drilled first. Six-inch hollow stem augers are used to produce an open hole through the overburden into the top of rock. Six-inch casing is then driven down to top of rock. The 6-inch casing is washed out using a roller bit which is also used to drill into the bedrock. This 5-5/8-inch roller bit provides a hole into the top of rock, where the 4-inch welded steel pipe will be seated. The 4-inch pipe is fabricated to required length and placed inside the 6-inch down to the bottom of the hole. The annulous between the 6-inch casing and the 4-inch casing provides a passage way for grout to be placed down the hole. As the 4-inch casing is grouted in-place, the 6-inch casing is removed from the hole.

Provided that the grout has not gotten into the 4-inch casing the Supervisory Geologists run a gamma log of the material. As the most permeable zone is determined, thus the location and depth of the well to the till is determined.

The 6-inch hollow stem augers are used to produce an open hole, the desired depth. The well point is fabricated and installed. A typical well point for this type of monitoring well is made up of a 2-foot sump, (sometimes 1-foot or 3-foot, per Supervisory Geologist), which has a welded cap at the bottom. Above the sump is welded a 10-slot stainless steel screen, (2" x 2' or 2" x 5'). Welded above the screen is a blank steel 2-inch pipe, which extends to within 0.5-feet of the existing grade. The well point is backfilled in-place with washed sand to a depth determined by Supervisory Geolgist. Bentonite is placed above the sand to prevent migration of contaminants into the sand. The well is backfilled with grout to within 3-feet of existing grade. A protective casing is then installed. A locking cap is fixed to each well be weld or thread.

Drilling activities resume at wells into the dolomite as the grout has set-up around the 4-inch casing. A 2-15/16-inch roller bit is used to drilled in to dolomite. This depth has been predetermined to be 5.0-feet, unless there is no water-bearing zone. If this is the case, the depth into rock shall be increased to 10.0-feet. If at this time no water-bearing zone has been encountered, the well shall be abandoned. Abandoned wells shall be plugged in accordance with specifications, established by the State of New York. Water producing wells will be provided with locking caps and protective casing.

As in all drilling activities, all cuttings and drilling water will be stored per contract specifications, until written instruction has been received by DCO.

TASK IV: Sediment Samples from Strata above Bedrock

Empire Soils Investigations, Inc., will collect sediment samples as requested by the Supervisory Geologist, with the concurrence of GCA. The method used will be hollow stem augers, drilled to desired depths and a split spoon will be driven and pulled. All samples will be surrendered to the Supervisory Geologists for classification and indications of significant changes in stratigraphy.

DOCUMENTATION OF DRILLING ACTIVITIES AND WELL INSTALLATION IN SUPPORT OF THE LOVE CANAL STUDY

Geological logs will be kept by each driller as each drill site is being drilled. Upon completion of each drilling activity the logs along with a quantity sheet will be turned over to the DCO - Empire Soils Investigations, Inc. The drilling logs will be used to varify drilling activities and the quantity sheets will be used to varify construction and installation of each well point. A copy of each shall be distributed to the Prime Contractor DCO and to the Supervisory Geologist DCO.

Empire Soils Investigations, Inc. - DCO will receive a copy of each of the following from the Supervisory Geologist DCO; work authorization, site sketch, well log, as-built drawing.

Weekly progress reports will be furnished by Empire Soils Investigations, Inc. - DCO, as required by contract.

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Page 5 of 5

DEMOBILIZATION

Upon completion of drilling activities, at each site, the following steps will be taken, to accomplish site restoration. The concrete vaults will be installed at each well location. Topsoil will be placed to repair any damage to lawns. Seed will be placed on all lawns where drilling has taken place. At the completion of these steps, the Supervisory Geologist will then sign the quantity sheets, and the site will be considered finished.

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Page 1 of 1

INFORMATION FLOW CHART

GENERAL SUPERINTENDENT
Vicky Anderson

QUALITY ASSURANCE/QUALITY CONTROL
Brian Schleifer

GENERAL FOREMAN
Jerry Malack

Technos, Inc.
QA Plan

Appendix Q
Plan 5.3

DRAFT

TECHNOS INC.

PROCEDURES FOR GEOPHYSICAL DATA AQUISITION AND CONTROL

(LOVE CANAL PROJECT)

I GENERAL DOCUMENTATION PROCEDURES

II COMPUTER PROCESSING OF DATA

III QUALITY ASSURANCE PROGRAM

- DATA CONTROL
- INSTRUMENT CALIBRATION
- CHAIN OF CUSTODY

Presented By
TECHNOS INC.
September 1980

I GENERAL DOCUMENTATION

--FORMAT FOR RECORDING AND SAFEGUARDING GEOPHYSICAL DATA--

(A) FIELD DATA

Field data relative to the project is recorded in bound Keuffel and Esser Co. (K and E) field notebooks. Separate notebook logs are maintained for each geophysical method. The location of all geophysical traverses and stations are recorded on the "standard" site map (1 inch = 200 feet) provided by GCA Corporation.

(B) GEOPHYSICAL DATA FORMAT (ON-GOING)

The data format for the respective methods as depicted in Table 1.

All recorded geophysical data is annotated and appropriate correlative information is entered into field notebooks on a daily basis. At the close of each working day all original records are collected and placed in the custody of an individual who is responsible for filing, allowing access to and safeguarding the records. In the case of the Love Canal Project, the Project Manager, R. C. BENSON, or a person designated in his absence is responsible for such data control.

Figure 1 depicts the internal document flow during on site data acquisition and analysis.

TABLE 1

DATA FORMAT FOR GEOPHYSICAL METHODS

METHOD	FORMAT
1. Ground Penetrating Radar (GPR)	Graphic Record
2. Electromagnetic (EM)*	Strip Chart
(a) Shallow (SEM)	"
(b) Deep (DEM)	"
3. Resistivity (RES)	Numerical reading recorded in field log book.
4. Metal Detector (MD)*	Strip Chart
5. Magnetometer (MAG)*	Strip Chart

---

*In the case of point-by-point measurements at respective stations numerical data or others results will be recorded directly into the respective field notebook.

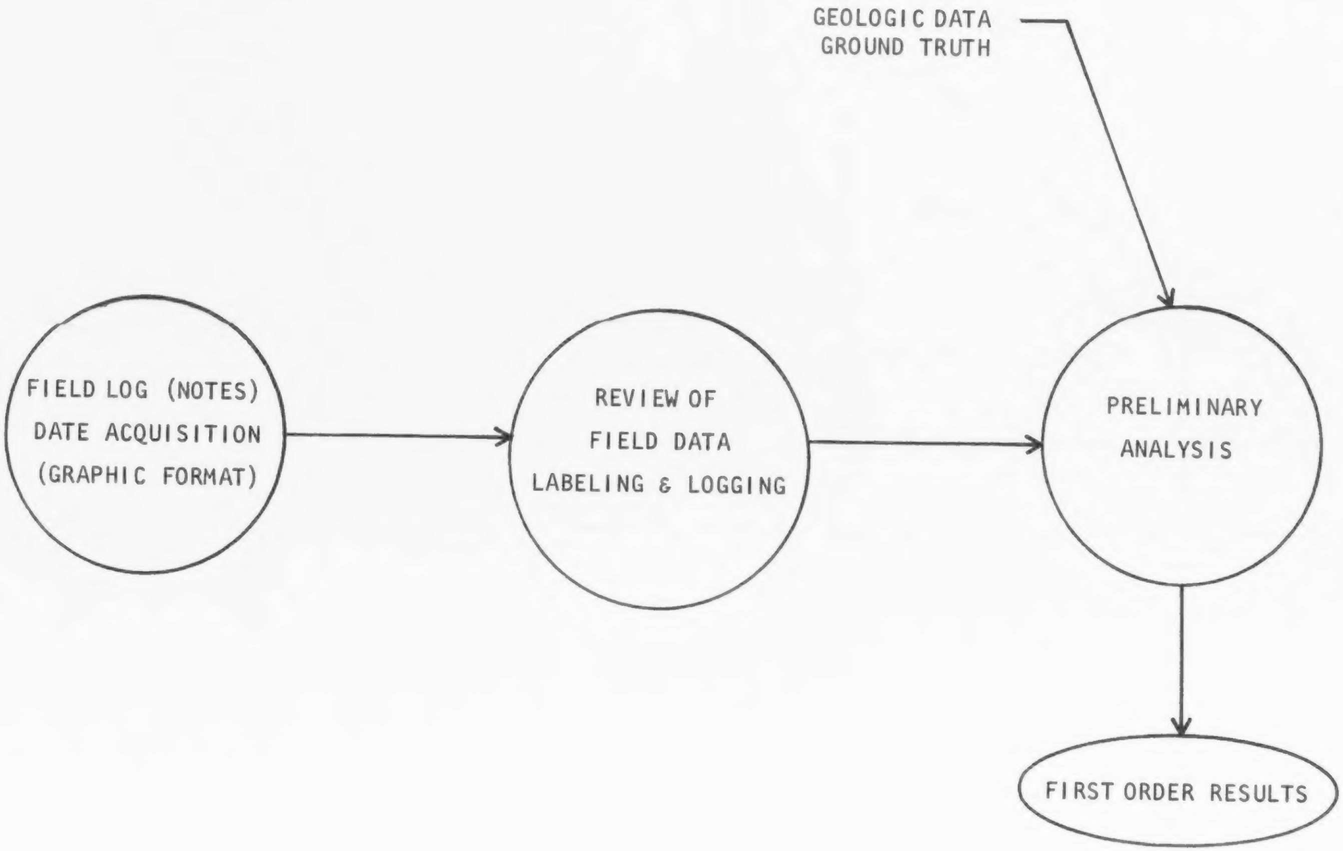
GEOLOGIC DATA
GROUND TRUTH
FIELD LOG (NOTES)
DATE ACQUISITION
(GRAPHIC FORMAT)
REVIEW OF
FIELD DATA
LABELING & LOGGING
PRELIMINARY
ANALYSIS
FIRST ORDER RESULTS

Figure 1. Technos Internal Document Flow During On Site Data Acquisition and Analysis.

II COMPUTER PROCESSING OF DATA

Figure 2 illustrates the flow of data in the field and office operation regime maintained by TECHNOS INC.

(A) Computer Processed Data - System Entrance

Data to be computer processed enters the system by means of one of two ways:

(1) the digitizer for strip chart records (EM, MAG AND MD) OR

(2) the keyboard for RES data.

(B) Computer Processing (Post-Entrance)

Computer processing may include the following:

- spatial corrections (compensating for variables in traverse speed);
- removal of "noise" indicative of cultural features (e.g. pipes and cables) is accomplished by the data analyst at time of entry;
- filtering of the data to remove short - period spatial variables;
- corrected and processed records;
- composite plots of records and
- three dimensional data plots (where appropriate).

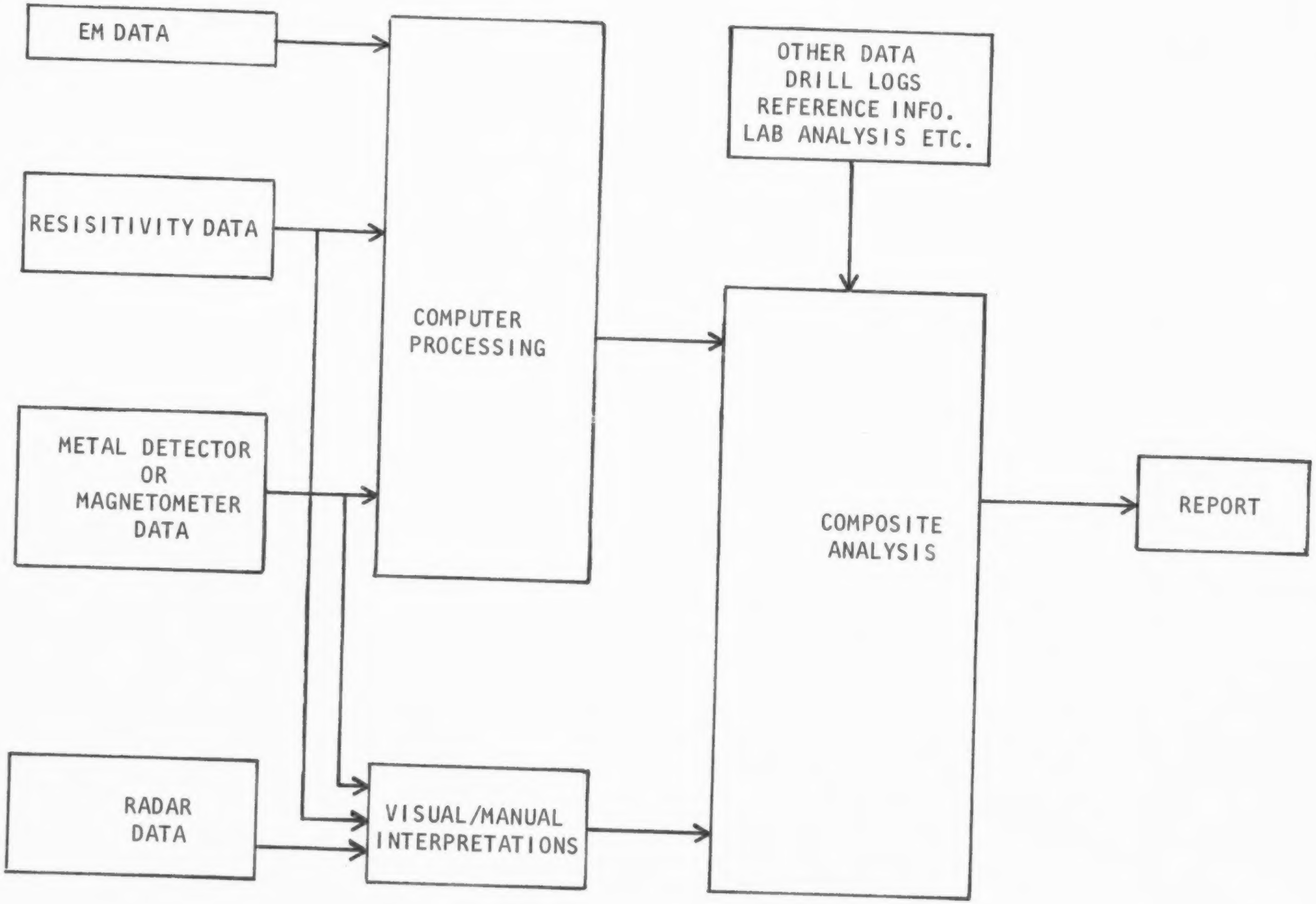
EM DATA
RESISITIVITY DATA
METAL DETECTOR OR MAGNETOMETER DATA
RADAR DATA
COMPUTER PROCESSING
VISUAL/MANUAL INTERPRETATIONS
OTHER DATA DRILL LOGS REFERENCE INFO. LAB ANALYSIS ETC.
COMPOSITE ANALYSIS
REPORT

Figure 2. Technos Internal Documentation Flow During Analysis in Home Office.

QUALITY ASSURANCE PROGRAM

DATA QUALITY CONTROL

Initial quality control of all data will be made by a professional geophysicist at the time of acquisition. Initial plotting of resistivity data will be done on station. This enables on-site evaluation of the raw data. All other data including GPR, MAG, or MD will be recorded on a graphic recorder or strip charts. This format offers a visual on-line means of initial quality control. Secondary quality control will occur as data is logged daily. Data are cross-checked at each step of analysis and processing. Final results are reviewed by two geophysicists to insure high "final quality" data interpretations.

INSTRUMENT CALIBRATION

Table 2 depicts the calibration techniques for documentation relative to respective techniques.

TABLE 2

Calibration techniques for geophysical instrumentation applied in Phase II of the Love Canal Project.

Instrumentation/Method	Calibration Details
1. GPR	Depth is presented as a electromagnetic propagation of time. All radar records are calibrated in time with an in-field 10 Nanosecond calibration standard.
2. EM	EM data is presented as apparent bulk conductivity. All systems are standarized at a known test site (near the TECHNOS INC. office facility in Miami, Florida). On-site tests are made on each traverse to check system linearity by recording both the zero and upper scale readings.
3. RES	Built-in system checks and standard "dummy loads" are used to check resistivity systems.
4. MD and/or MAG	Both of these systems provide relative readings. The only calibration required is a systems sensitivity test which is made each time these instruments are used.

CHAIN OF CUSTODY

The previous section General Documentation describes the means by which the geophysical data (on-going) is acquired and safeguarded.

Upon completion of field work all original data including log books and related information shall be safeguarded in locked file cabinets located on the property of TECHNOS INC.

All original data acquired in this project shall be retained by TECHNOS INC. through the "FINAL REPORT PHASE" of this effort. Only Xerox copies of the original data will be transferred as part of the required reporting.

Distribution of the TECHNOS INC. FINAL REPORT on the Love Canal Project will be determined by GCA Corporation.

Appendix Q
Plan 5.4

GeoTrans, Inc.
QA Plan

1.0 PROJECT DESCRIPTION AND ORGANIZATION

1.1 GENERAL

The purpose of this project is to model the ground-water flow at the Love Canal site. This includes the shallow saturated zone in the unconsolidated strata and the deeper aquifer in the Lockport dolomite. Existing or easily modified numerical models will be applied to address the significance of areal hydrologic changes, existing and potential pollution control methods, and swales and possible man-made avenues of ground-water movement.

The modeling activities will be coordinated with drilling and data collection activities. The models will be based on the data obtained from the site. Additionally, the models will be applied to assist in the selection of drilling sites and hydrologic testing.

The models will be calibrated with the site data in a consistent manner. The calibrated models will be used to make predictions for the spread of contamination over the next five years. The predictions will consider conservative contaminants only in the ground water under varying conditions of discharge and recharge.

1.2 ORGANIZATION

The modeling activities outlined in the previous section can be grouped into five tasks for planning purposes.

These are: 1) initial site visit, assessment of available data and construction of a preliminary model; 2) analysis of new data, incorporation into models and calibration of models; 3) a final predictive and sensitivity analysis; 4) preparation of the final report describing the finding of this study; and 5) quality assurance. The project organization (including names of key personnel) is shown in figure 1.

1.3 TASK DESCRIPTIONS

1.3.1 Task 1

The first phase of the study requires the construction of preliminary models that can be used to aid in well siting and data collection design. In order to construct such a model it is necessary to visit the site, consult with the primary contractor and EPA, to assess existing information, and to select appropriate models. As previously indicated, we anticipate using only well documented existing computer codes. We see no need for model development -- it can be very expensive and cause major delays. In addition to selecting a particular model (Intercomp, 1976; Trescott and Larson, 1976; and Trescott, Pinder, and Larson, 1976), at this point it will also be necessary to determine whether the aquifers should be modeled separately. If the aquifers are modeled separately, two-dimensional area models may

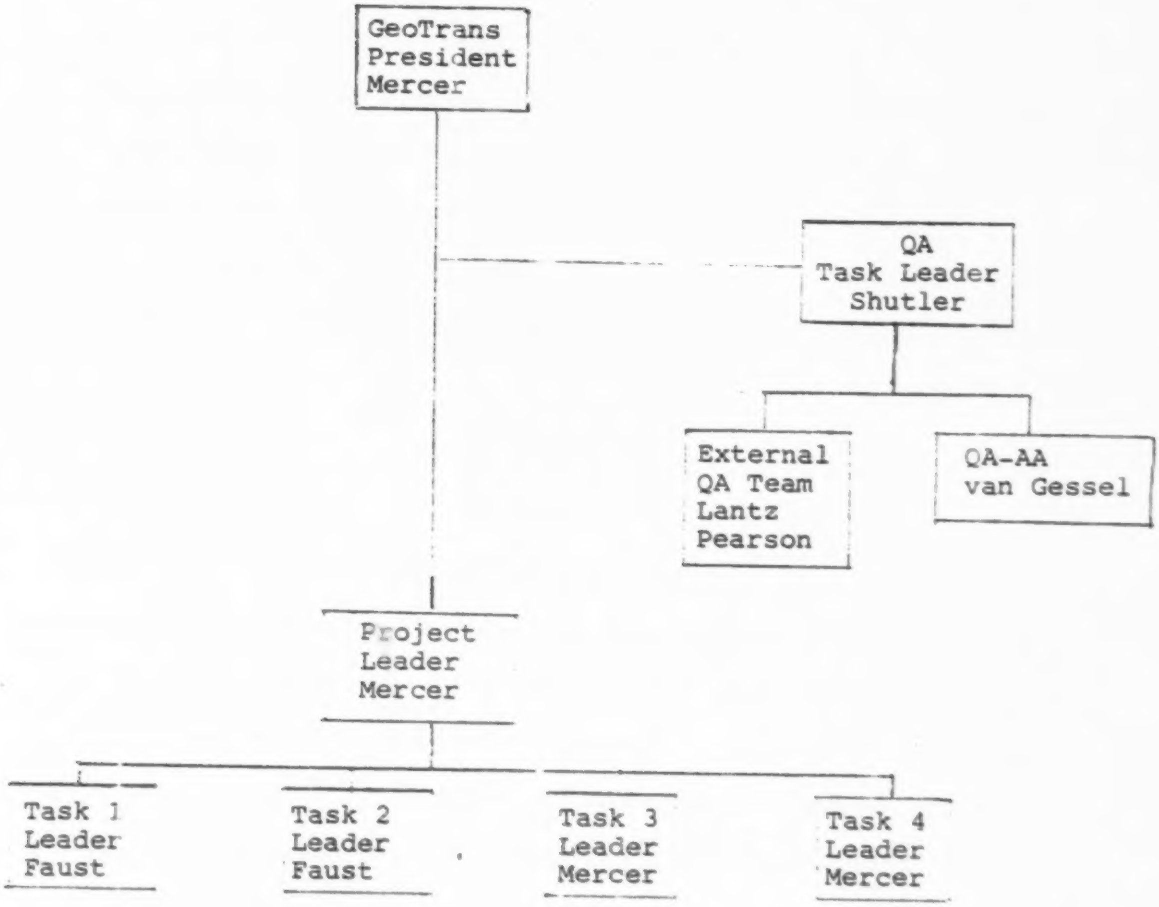
GeoTrans
President
Mercer
QA
Task Leader
Shutler
External
QA Team
Lantz
Pearson
QA-AA
van Gessel
Project
Leader
Mercer
Task 1
Leader
Faust
Task 2
Leader
Faust
Task 3
Leader
Mercer
Task 4
Leader
Mercer

Figure 1. Project organization chart.

be used. Three-dimensional area models will be required if they are modeled simultaneously. Once the initial model is selected, it will be used in conjunction with existing data to aid in data collection design.

1.3.2 Task 2

Analysis of the data and data reduction, as well as updating the preliminary models will take place as long as data are being collected. Updating of the preliminary model is often referred to as history matching or model calibration. It involves comparing the results from the model with field observations of water levels. Estimated hydrologic parameters are adjusted in a consistent manner to minimize the discrepancies between model results and field data.

1.3.3 Task 3

During this task, sensitivity and predictive analysis will be performed with the ground-water flow model(s) that incorporate all available data. The predictive analysis will indicate potential movement of the contamination plume during the next five years. The prediction will account for ground-water convection of the constituents, but neglect dispersion and sorption.

1.3.4 Task 4

The final draft report will be written during the period November 1 to November 15, 1980 by Drs. Faust and Mercer. A final report will be submitted within seven days after receipt of comments by the prime contractor.

1.3.5 Task 5

Because of the sensitivity of the Love Canal contamination problem, quality assurance must receive special attention in this study. Consequently, we have made quality assurance a separate task. This task involves specification of a project quality assurance plan with input from the prime contractor and EPA; internal documentation of model modifications, basic assumptions, and hand calculations; and external review.

2.0 MAJOR PROJECT ELEMENTS

From the task descriptions, it is seen that there are four major elements that are concerned with Quality Assurance. These are:

- model selection
- model modification
- model application
- reporting

Each of these are discussed in more detail in section 3.

3.0 QA OBJECTIVES

3.1 GENERAL

The overall objective of the GeoTrans' QA program is to assure that the models it develops, the applications it performs, and the reports that it delivers are of sound technical and scientific quality, are usable, satisfy contract requirements, and are supported by an adequate record of their evolution.

3.2 SPECIFIC OBJECTIVES

The specific objectives of the GeoTrans' QA procedures include:

- Verification - through cross-checks, debugging procedures, and model tests, assure that the model satisfies the model design specifications.
- Validation - through model tests and application to known laboratory or field situations, assure that the model accurately satisfies the performance specifications.
- Documentation - through a system of controls and documentation, assure that the mechanical and logical evolution of the model and/or its application beyond the first baseline can be tracked and evaluated, that the nature, characteristics, and validity of the model and/or its application are readily discernible, and that the final report is consistent with the work performed.

- Accountability - through a system of controls and a chain of documentation, assure that the persons responsible for any action materially and directly affecting the development, alteration, demonstration, evaluation or application of the model or preparation of the final report are identified with their actions and the rationale therefore.

- Integrity - through a system of controls, documentation, audits and reviews, assure that verification and validation of the models are accurate and complete, that baselines are protected from inadvertent or undocumented change, that nonconformances and errors are recorded and corrected, that the final report accurately reflects the project record, and that all other aspects of the QA program are satisfied.

3.3 PROJECT TASK QA OBJECTIVES

Of the five project tasks, four involve quality assurance procedures, the remaining task being the implementation of the QA controls. In the first task preliminary data and published reports are reviewed and appropriate models are then selected. The chosen models must then be adapted to project needs and computer facilities. QA checks are required to review:

- adequacy of literature review
- appropriateness of model selection
- reliability of model modifications

The second task includes the analysis of field data and its incorporation into the models. This task requires checks on:

- calculations used in data analysis
- conceptual basis for models of site
- model input data

The third task involves the final applications of the models to the site conditions. For this task, reviews should be done on:

- model input data
- credibility of predicted results
- adequacy of overall modeling approach

Associated with each of the first three tasks and the fourth are written reports. Each written communication to the client from this office must be checked for assured technical and scientific quality. Such reports include:

- Topical memos to GCA
- Biweekly progress reports
- The ground-water model procedures manual
- The final report

3.4 REFERENCES

This Project QA Plan outlines the specific QA procedures to be used in this study. GCA has a copy of the GeoTrans QA plan that describes the system of controls, audits, and reviews applicable to the general technical work done by GeoTrans. The Program QA Plan also defines terms and abreviations routinely used in QA documents.

3.5 QA CONTACTS

The primary contact for QA on this project is the director of QA, Norman D. Shutler, Deane, Snowden, Shutler, Garrish, & Gherardi, phone # (202)462-0561. The QA administrative assistant is Virginia van Gessel, GeoTrans, Inc., phone # (703)435-4400.

4.0 DOCUMENT CONTROL PROCEDURES

GeoTrans' quality assurance plan for document control has two aspects relating to: 1) Document movement procedures, and 2) Storage procedures.

All documents leaving or entering our offices are entered in a log which identifies the originator, the date received or sent, the project code, and identifying document code. GeoTrans will use GCA's suggested document codes (in addition to GeoTrans' own numbers) from Table 4-2 in GCA's QA Project Plan. All documents will be identified by the Love Canal Project code (LC1-619-026), the Subcontractor code (14), the Document code (alpha identity), and Serial number.

GeoTrans maintains a project QA documentation file on each of its projects identified by a specific internally assigned project number and file identifiers. Control Identification Numbers (CINs) are used to identify all project materials, including reports and software. They consist of up to three numeric-alpha pairs as in 91A-00M-06A. The first number refers to the project number which is the same as that used for accounting purposes. The associated alpha character refers to the project task. The second number is used to identify a particular model that is used for the given project and task. If no model is involved, 00 is used. The associated alpha character for this pair refers to the following key:

A - Performance Specifications
B - Design Specifications
C - Code
D - Test Specifications
E - Test/Application Data
F - Test/Application Results
G - Reports
H - Problems/Corrective Action
I - Reviews
J - Audits
K - Final Report
L - Project Log Book
M - Memorandum
P - Photos, Maps, Drawings
Q - Project QA Plan

The third number, in the case of data, refers to the particular set. For written material such as memoranda, it refers to their sequence number. The associated alpha character allows for revisions to be tracked. It should be noted that some of the characters may be omitted. For example, if no project task is involved, the first alpha character may be omitted. If there are no revisions, the final alpha character may be omitted. In the case of a code, the third number will also be 01, and therefore may be omitted. Given this information, it may be determined that the example given above refers to the first revision of memorandum number 6 regarding task A of project 91. As another example, 91C-01C-A refers to revision A of code one used in task C of project 91.

Materials are filed daily in chronological order in the appropriate file utilizing the corresponding code identification number from the classification system shown above. This thorough and orderly storage of documents assures easy retrievability and a comprehensive record of all communication covering every aspect of this project.

5.0 CHAIN OF CUSTODY PROCEDURES

For this project, chain of custody procedures are not necessary, because GeoTrans will not have custody of any samples.

6.0 PROJECT QA PROCEDURES

6.1 GENERAL

The specific objectives of the GeoTrans' QA plan (Verification, Validation, Documentation, Accountability, and Integrity) are achieved by a system of controls, audits, and reviews. For this project the most formal level of quality assurance is appropriate. This involves formal external reviews and audits.

For this project publicly available models that have been well verified and validated will be used. Verification of model changes will be accomplished by running the example problems given in the original model documentation. Because these models have already been validated, reference to previous applications will be sufficient.

Documentation of QA action is maintained by the QA administrative assistant - Virginia van Gessel. For this project, records will be maintained for the following baselines:

- The Project QA Plan
- The model design and performance specifications
- Model modifications
- Computer codes
- Data used for code testing or for applications
- The results of applications
- Problems that occur when using models
- Remedial actions taken to correct problems

- The final draft report
- Audits and reviews

Accountability is accomplished by identifying those persons responsible for any action materially affecting the reliability of a model, application, or report. The reason for the action must also be stated. The documentation of various actions includes a cover page clearly identifying the responsible persons and their rationale.

Integrity is maintained by the controls and documentation already mentioned and by reviews and audits. For this project, an external review team (Drs. Lantz and Pearson of Intera, Inc., Houston) will review all baselines. This review is in addition to the internal reviews performed informally. Two audits to assure that the QA procedures are being properly implemented will be performed by Dr. Shutler of Deane, Snowden, Shutler, Garrish, & Gherardi. Dr. Shutler will also serve as leader of the QA task of this project in order to maintain more direct control of QA on this project.

An overview of the major project phases, preliminary outputs, and resulting baselines is shown in table 1. A Summary of the various planned review and audits is given in table 2. Table 3 indicates the level of review for the specific QA concerns listed in section 3.

Table 1. Major project phases - description, preliminary output, and baselines.

Project Phase	Model Selection	Model Modification	Testing	Model Application	Final Acceptance
Description	Document perf. Spec., review avail. models, select models.	Acquire models, modify, compile user manual, design tests.	Test, evaluate.	Perform study, prepare draft report.	
Preliminary Output		User manual		Final report	
Baseline	Performance specs.	Test specs.	User manual, test data, test results.	Model data, model results.	Final report

Table 2. Summary of QA reviews for project.

Review Type	Material or actions reviewed	Reviewer
Informal Internal Review	On all preliminary output and baselines	GeoTrans personnel
Formal External Review	On all baselies scheduled for 10 Oct 1980 and 17 Nov 1980	R.Lantz F.pearson (Intera)
QA Audit	Scheduled for 10 Oct 1980 and 26 Nov 1980	N.Shutler

Table 3. QA reviews for specific project material and actions.

Specific material or action	Informal	Formal
Adequacy of literature review	xx	xx
Model selection	xx	xx
Model modifications	xx	xx
Calculations used	xx	
Conceptual basis of models	xx	xx
Model input data - prelim.	xx	
Model input data - final	xx	xx
Credibility of predictions	xx	xx
Overall modeling approach	xx	xx
Topical memos to GCA	xx	
Biweekly progress reports	xx	
Ground-water model manual	xx	xx
Final report	xx	xx

7.0 CORRECTIVE ACTION PROCEDURES

7.1 GENERAL

Problems requiring corrective action may become apparent in several ways:

- a model user may encounter or discover a problem with a previously baselined code,
- an informal reviewer may note a problem with a model, its application, or interpretation of its results,
- a formal reviewer may note a problem with a baseline,
- an auditor may note a problem with the implementation of QA procedures.

When problems are encountered with baselined models, a problem report document is initiated. Remedial action is then taken to correct the problem. The problem may be corrected by either modifying the model to eliminate the problem or by changing the model specifications. These require baseline changes. In some cases the current version of the model may have a problem that was the result of an inadvertent or unauthorized change. In this situation the problem can be resolved by reverting to the original baselined model and discarding the incorrect model.

Informal reviews of project work may point out problems to the person performing the work. No formal documentation is required for this type of review, but the problem should be resolved as soon as possible.

Formal reviews and audits require written findings and recommendations. The QA-AA shall monitor all formal reviews and audits, and shall initial a problem report for any major problems, and for any minor problems not corrected within 48 hours.